

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of Grubb

Application Serial No. 10/602,945

Filing Date: June 24, 2003

Art Unit 1795

Examiner Tracy Mae Dove

Confirmation No. 1816

FERRITIC STAINLESS STEEL
HAVING HIGH TEMPERATURE
CREEP RESISTANCE

Docket No. RL-1627DIV

VIA ELECTRONIC MAIL

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APPEAL BRIEF

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Allegheny Technologies Incorporated, the parent company of ATI Properties, Inc., the assignee of the above-referenced patent application (the "Subject Application"), submits this appeal brief in accordance with the provisions of 37 C.F.R. § 41.37 in response to (i) the Final Office Action mailed on June 5, 2008, (ii) the Notice of Appeal filed on December 5, 2008, and (iii) the Notice of Panel Decision from Pre-Appeal Brief Review mailed on December 23, 2008.

The Commissioner is hereby authorized to charge PTO Deposit Account No. 11-1110 for any fees necessary for consideration of this brief and appeal, including the fee under 37 C.F.R. § 41.20(a)(2) for filing a brief in support of an appeal, and the fee under 37 C.F.R. § 1.17(a)(5) for the petition under 37 C.F.R. § 1.136(a) for a five (5) month extension of time filed concurrently with this appeal brief.

I. REAL PARTY IN INTEREST

The real party in interest is Allegheny Technologies Incorporated, by reason of assignment of the parent application (Serial No. 09/998,487, U.S. Patent No. 6,641,780) of the Subject Application and the invention from the inventor to ATI Properties, Inc., a wholly-owned subsidiary of Allegheny Technologies Incorporated, recorded at Reel 012345, Frame 0096.

II. RELATED APPEALS AND INTERFERENCES

A Notice of Appeal and a Pre-Appeal Brief Request for Review were filed in the Subject Application on December 5, 2008. The Pre-Appeal Brief Conference resulted in a Notice of Panel Decision from Pre-Appeal Brief Review mailed December 23, 2008 in which the Conference Panel upheld the rejections in the June 5, 2008 Final Office Action. The Conference Panel passed the appeal proceeding to the Board of Patent Appeals and Interferences (the "Board") for full consideration.

Appellant is not aware of any other appeals or any interferences that may be related to, may directly affect or be directly affected by, or have a bearing on the decision of the Board in the present appeal.

III. STATUS OF CLAIMS

A. Claim Status

Claims 36-40, 42, and 43 are pending in the Subject Application. The Subject Application is a divisional application of United States Patent Application Serial No. 09/998,487, filed November 30, 2001, which issued as United States Patent No. 6,641,780 on November 4, 2003.

The Subject Application was originally filed with claims 1-39. Claims 1-22 were canceled without prejudice or disclaimer in a Preliminary Amendment filed on June 24, 2003. Claims 40-54 were added in a Supplemental Preliminary Amendment filed on September 18, 2003. Claim 41 was canceled without prejudice or disclaimer in a Response to Office Action filed on June 21, 2005. Claims 23-35 and 44-54 were canceled without prejudice or disclaimer in a Response to Office Action filed on November 22, 2006.

Claims 36-40, 42, and 43 were under examination on the merits and form the basis of this Appeal. Claim 36 is the only independent claim.

B. Procedural Posture

The Subject Application has had a relatively long prosecution history that includes seven (7) Office Actions and two (2) Requests for Continued Examination. The sixth Office Action (non-final) in the Subject Application was mailed February 29, 2008, signed by primary Examiner Tracy Dove. At some point thereafter, Examiner Dove apparently went on leave from the Office. An interview was held between Appellant's representative and Supervisory Patent Examiner (SPE) Patrick Ryan during Examiner Dove's leave. An agreement was reached between Appellant's representative and SPE Ryan regarding the allowability of claims 36-40, 42, and 43. Appellant filed a response on February 29, 2008 based on the agreement reached with SPE Ryan.

Responsive to Appellant February 29, 2008 response, Examiner Dove, apparently back from leave, issued a Final Office Action mailed June 5, 2008. Examiner Dove apparently denied any knowledge of and participation in the agreement with SPE Ryan. Currently, the putative agreement regarding the allowability of claims 36-40, 42, and 43 is not acknowledged by Examiner Dove.

In the Final Office Action mailed on June 5, 2008 (the "Final Office Action"), pending claims 36-40, 42, and 43 were rejected by the Examiner under 35 U.S.C. § 103(a) as allegedly having been obvious over United States Patent No. 6,613,468 to Simpkins et al. ("Simpkins") in view of Japanese Patent Application Publication No. 2000-294256 to Tarutani et al. (referred to throughout the prosecution of the Subject Application as "Taruya"), as evidenced by United States Patent No. 5,424,144 to Woods et al. ("Woods").

In response to the Final Office Action, Appellant filed a Notice of Appeal and a Pre-Appeal Brief Request for Review on December 5, 2008. The Conference Panel issued a Notice of Panel Decision from Pre-Appeal Brief Review on December 23, 2008 indicating that the Subject Application remains under appeal because issues remain for consideration by the Board.

Accordingly, claims 36-40, 42, and 43 stand rejected and are the subject of the present appeal. The text of claims 36-40, 42, and 43 is set forth in the Claims Appendix beginning at page 29 of this Appeal Brief.

IV. STATUS OF AMENDMENTS

All amendments have been entered and the current text of the pending claims is presented in the Claims Appendix.

V. SUMMARY OF CLAIMED SUBJECT MATTER

All references herein to the specification of the Subject Application refer to the page and line numbers of the specification as originally filed, not to the paragraph numbers of the as-published version of the Subject Application. The claims under consideration in the present appeal include one (1) independent claim, claim 36.

Independent claim 36 recites a solid oxide fuel cell comprising:

an anode;

a cathode;

an electrolyte comprising stabilized zirconia, wherein the electrolyte is intermediate the anode and the cathode; and

an interconnect providing a current pathway from the anode, the interconnect comprising a ferritic stainless steel comprising:

greater than 25 weight percent chromium,

0.75 up to 1.5 weight percent molybdenum,

up to 0.05 weight percent carbon, and

at least one of niobium, titanium, or tantalum, wherein the weight percentages of niobium, titanium, and tantalum satisfy the equation

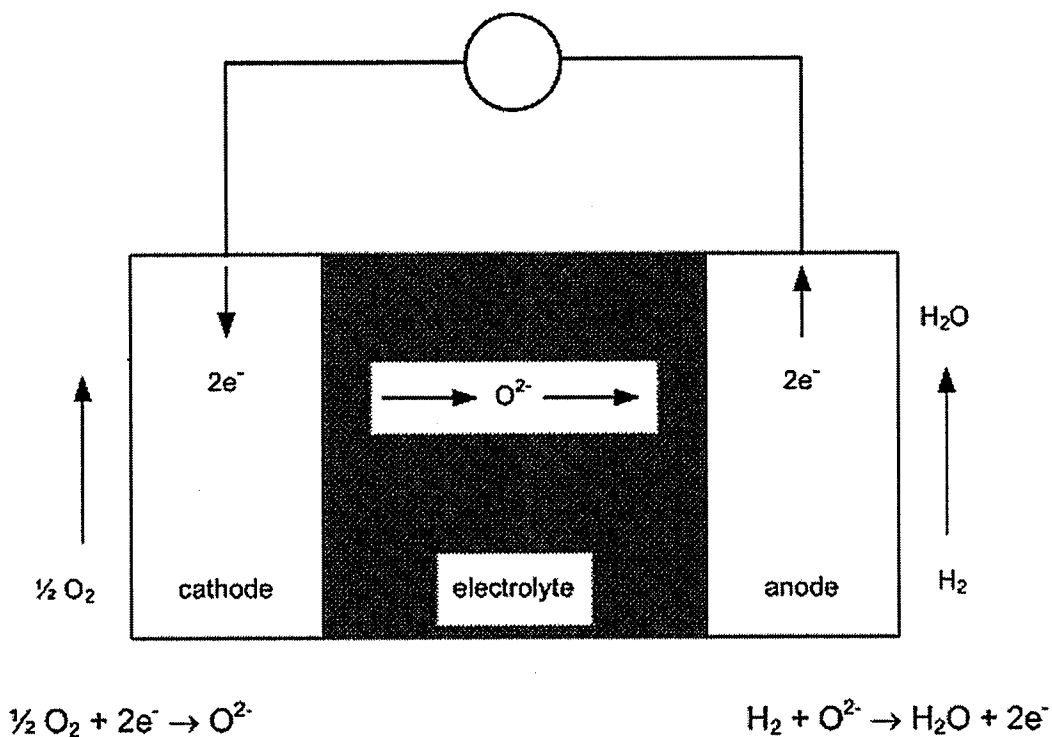
$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1,$$

wherein the steel has a coefficient of thermal expansion within about 25 percent of the coefficient of thermal expansion of stabilized zirconia between 20°C and 1000°C and exhibits at least one creep property selected from the group consisting of creep rupture strength of at least 1000 psi at 900°C, time to 1% creep strain of at least 100 hours at 900°C under load of 1000 psi, and time to 2% creep strain of at least 200 hours at 900°C under load of 1000 psi.

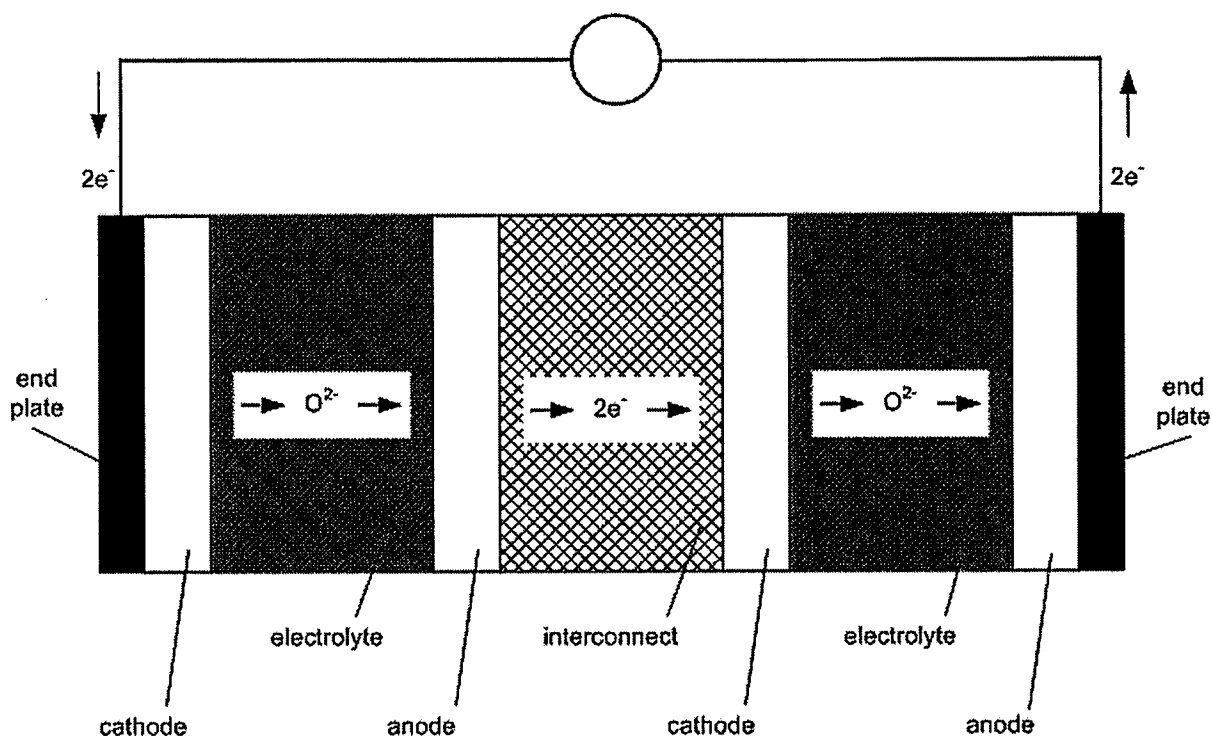
Solid oxide fuel cells (SOFCs) generate electric power through electro-chemical reactions. A single SOFC comprises an anode and a cathode separated by a solid oxide ceramic electrolyte, which is typically a form of stabilized zirconia, such as, for example, yttria-stabilized-zirconia (YSZ). The solid oxide electrolyte divides a SOFC into a fuel side and an oxidant side. During operation, the fuel side contains a fuel fluid, such as, for example, methane, hydrogen, petroleum-based fuels, coal gas, and the

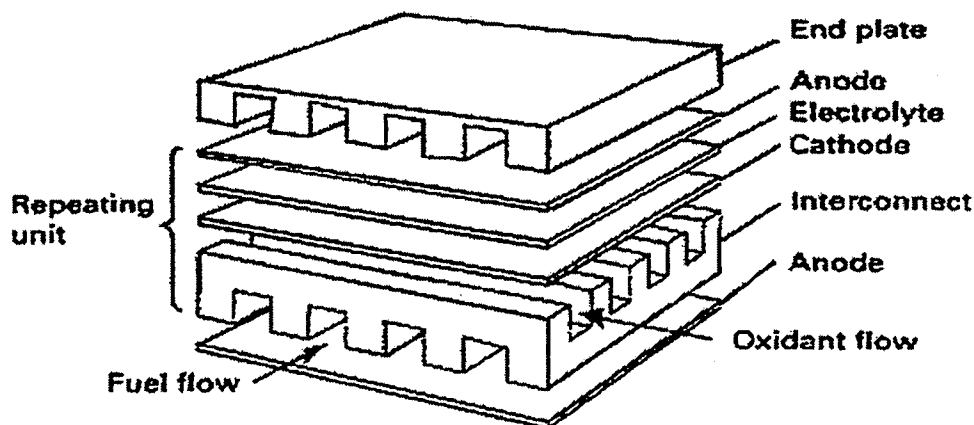
like, which are chemically capable of being oxidized. During operation, the oxidant side contains an oxidant, such as, for example, oxygen or oxygen-containing gas mixtures (e.g., air), which are capable of being reduced. Generally, the fuel side corresponds to the anode of a SOFC and the oxidant side corresponds to the cathode of a SOFC.

In operation, a fuel is oxidized (e.g., combusted) at the anode where it releases electrons. The electrons transport through an external circuit to the cathode. Molecular oxygen (O_2) is reduced at the cathode by the electrons from the external circuit, forming oxide anions (O^{2-}). The solid oxide electrolyte is permeable to the oxide anions, which transport through the electrolyte from the cathode to the anode. The combined ionic flow through the electrolyte and the electrical flow through the external circuit (both charge flows) completes the electro-chemical circuit. This process is illustrated below with molecular hydrogen (H_2) as the fuel, for example.



Useful energy is extracted from a SOFC by using the electrons transporting through the external circuit to do work. However, the amount of energy available from a single SOFC is limited. Hence, planar SOFC stacks have been developed. Planar SOFCs comprise two or more single SOFCs stacked together in series. The individual SOFCs comprising a planar SOFC stack are separated by structures termed interconnects. Interconnects function to collect and transport the electrons generated by the electrochemical reactions in the individual fuel cells. Interconnects also function as a physical separator for the fuel streams and the oxidant streams associated with the anodes and cathodes, respectively. A planar SOFC stack comprising two SOFC in series is illustrated below.





The material used to form SOFC interconnects must be electrically conductive to complete the electrochemical circuit. In addition, because interconnects are positioned adjacent to anodes and cathodes in SOFCs, the material used to form interconnects must be oxidation resistant, thermally stable, and mechanically stable.

SOFCs operate over a wide range of temperatures. For example, at start-up a SOFC may be around room temperature (20°C). During operation, the temperature of a SOFC may reach temperatures upwards of 1000°C. In addition, temperature fluctuations during operation may expose the materials forming SOFCs to substantial thermal cycling. As a result, it is important that the material used to form interconnects have thermal expansion properties, such as, for example, the coefficient of thermal expansion (CTE), substantially matching those of the materials used to form the other components of the SOFC, such as anodes, cathodes, and electrolytes, which contact or are in very close proximity to the interconnect.

As mentioned above, SOFC electrolytes are typically formed of solid oxide ceramics, such as YSZ or other stabilized zirconia. SOFC anodes and cathodes are also commonly formed from ceramic or ceramic-metal composite (cermet) materials. Accordingly, SOFC interconnects have previously been fabricated from ceramic materials that are electrically conductive at SOFC operating temperatures, for example, lanthanum chromite (LaCrO_3) doped with calcium oxide (CaO) or strontium oxide (SrO).

However, doped ceramic materials are brittle, expensive, and poor electrical conductors, particularly when compared to metals and metal alloys.

Fabricating SOFC interconnects from stainless steels provides certain advantages because steels have relatively good electrical conductivity and are less brittle than ceramics. However, in interconnect applications; stainless steels may exhibit certain undesirable properties such as relatively high levels of oxidation, thermal expansion, and creep. Nevertheless, the present inventor discovered that producing a ferritic stainless steel comprising certain compositional features will result in a material that will function exceptionally in SOFC interconnect applications.

The present inventor discovered that a ferritic stainless steel comprising greater than 25 weight percent chromium, 0.75 up to 1.5 weight percent molybdenum, up to 0.05 weight percent carbon, and at least one of niobium, titanium, or tantalum may be used to form an interconnect that performs advantageously in SOFC applications. Specification, page 10, line 14 - page 11, line 2. The ferritic stainless steel exhibits thermal expansion properties that substantially match the thermal expansion properties of ceramic SOFC materials like stabilized zirconia. In particular, the ferritic stainless steel is characterized in that it has a CTE within about 25 percent of the CTE of stabilized zirconia between temperatures of 20°C and 1000°C. Specification, page 11, lines 8-13,

In addition, the ferritic stainless steel recited in claim 36 exhibits significantly improved high temperature mechanical properties relative to prior ferritic stainless steels. The present inventor discovered that a ferritic stainless steel comprising at least one of niobium, titanium, or tantalum, characterized in that the weight percentages of niobium, titanium, and tantalum satisfy the equation:

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1,$$

possesses significantly improved high temperature mechanical properties.

Specification, page 15, line 1 - page 16, line 2. In particular, the ferritic stainless steel is

characterized in that it exhibits certain exceptional creep properties. The ferritic stainless steel exhibits either (i) a creep rupture strength of at least 1000 psi at 900°C, (ii) a time to 1% creep strain of at least 100 hours at 900°C under a load of 1000 psi, and/or (iii) a time to 2% creep strain of at least 200 hours at 900°C under a load of 1000 psi. Specification, page 11, lines 3-7.

Creep is defined as strain (i.e., deformation) occurring under stress (i.e., applied or induced load) over time. Creep of SOFC interconnects may occur at the high temperatures encountered during operation of SOFCs. The development of creep strain in SOFC interconnects can cause a loss of cell integrity (i.e., a breach between the interconnected cell components), which can result in the leakage of gaseous fuel and oxidant. The interconnect comprising the ferritic stainless steel recited in claim 36 has been shown to significantly improve creep resistance. Specification, page 28, line 10 - page 39, line 5.

The described ferritic stainless steel has improved microstructural stability, enhanced mechanical properties, and greater resistance to creep at high temperatures relative to prior ferritic stainless steels as demonstrated in the testing presented on pages 23 through 44. Accordingly, the interconnect recited in claim 36 comprising the described ferritic stainless steel exhibits improved microstructural stability, enhanced mechanical properties, and greater resistance to creep at high temperatures. As a result, the SOFC recited in claim 36 represents a significantly improved SOFC.

VI. GROUND OF REJECTION TO BE REVIEWED ON APPEAL

1. Whether the Examiner has established a *prima facie* case that claims 36-40, 42, and 43 are unpatentable under 35 U.S.C. § 103(a) as having been obvious over Simpkins in view of Taruya, as evidenced by Woods. Appellant submits that the Examiner did not establish a *prima facie* case of obviousness.

2. Whether, if the Examiner did establish a *prima facie* case that claims 36-40, 42, and 43 are unpatentable under 35 U.S.C. § 103(a) as having been obvious over Simpkins in view of Taruya, as evidenced by Woods, the record evidence of unexpected results nevertheless rebuts the *prima facie* case. Appellant submits that the preponderance of the record evidence supports the nonobviousness and patentability of claims 36-40, 42, and 43.

VII. ARGUMENT

(A) THE EXAMINER HAS NOT ESTABLISHED A *PRIMA FACIE* CASE THAT CLAIMS 36-40, 42, and 43 ARE UNPATENTABLE UNDER 35 U.S.C. § 103(a).

Obviousness under 35 U.S.C. § 103(a) is a question of law based on at least three underlying findings of fact:

- (1) The scope and content of the prior art;
- (2) The differences between the claimed invention and the prior art; and
- (3) The level of ordinary skill in the pertinent art.

Based on these facts, the legal conclusion of whether a claim, as a whole, is obvious or non-obvious is made based on a preponderance of the evidence standard. See Graham v. John Deere Co., 383 U.S. 1, 17-18 [148 USPQ 459] (1966); KSR International Co. v. Teleflex Inc., 550 U.S. 398 [82 USPQ2d 1385] (2007); In re Oetiker, 977 F.2d 1443 [24 USPQ2d 1443] (Fed. Cir. 1992).

To this end, the MPEP provides that the contents of a § 103(a) rejection set forth in an Office Action should include:

- (1) the relevant teachings of the prior art relied upon, preferably with reference to the relevant column or page number(s) and line number(s) where appropriate;
- (2) the difference or differences in the claim over the applied reference(s);
- (3) the proposed modification of the applied reference(s) necessary to arrive at the claimed subject matter; and
- (4) an explanation as to why the claimed invention would have been obvious to one of ordinary skill in the art at the time the claimed invention was made.

MPEP § 706.02(j).

As part of the determination of the scope and content of the prior art, prior art references must be considered in their entirety, *i.e.*, as a whole, including portions that

would lead away from the claimed invention. MPEP § 2141.02(VI). As part of the determination of the differences between the claims and the prior art, all of the words and features recited in the claims must be considered in judging the patentability of the claim against the prior art. MPEP § 2143.03. Indeed, in determining the differences between the prior art and the claims, the question under § 103(a) is whether the claimed invention as a whole would have been obvious. MPEP § 2141.02.I. It is the invention as a whole, and not some part of it, which is evaluated for obviousness under § 103. MPEP § 2141.02.V.

Accordingly, a determination regarding the obviousness or non-obviousness of the claims in a patent application involves a direct comparison of the subject matter of the claims, as a whole, to the teachings of the cited references, as a whole. A *prima facie* case of obviousness requires that the claims would have been obvious to a person skilled in the art at the time of the invention despite the differences between the claims and the teachings of the cited references. Thus, rejections on obviousness grounds cannot be sustained with mere conclusory statements or unsupported assertions. The Examiner must clearly communicate logical reasoning with rational underpinnings based on a preponderance of factual evidence to support the legal conclusion of obviousness. See MPEP § 2141.

Appellant respectfully submits that the Examiner has failed to properly establish a *prima facie* case under § 103(a). In the present matter, there are very significant differences and substantial distinctions between the SOFC recited in the present claims and the subject matter disclosed in Simpkins, Taruya, and Woods. These differences and distinctions create a very large gap between the prior art and the claimed invention that is "so great as to render the [present claims] nonobvious to one reasonably skilled in the art." MPEP 2141.III (quoting Dann v. Johnston, 425 U.S. 219, 230 [189 USPQ 257, 261] (1976)).

The primary reference relied upon by the Examiner in rejecting the present claims is Simpkins. Simpkins discloses the general physical structure of SOFCs.

Simpkins, 2:61-3:9. Simpkins discloses the individual structures that comprise a SOFC, including anodes, cathodes, electrolytes, and interconnects, and the manner in which these individual structures are assembled to form a SOFC. Id. Simpkins also discloses mat and spacer assemblies that are described as being positioned between the electrochemical cells (i.e., the anode-electrolyte-cathode assemblies) and the interconnects in a SOFC stack. Simpkins, 3:10-20. The mat and spacer assemblies comprise a porous gas diffusion mat (comprising matted wire, fibrous material, or other mesh material) positioned on the side of a spacer. Simpkins, 5:1-21.

The mat and spacer assemblies are described in Simpkins as being relatively flexible structures functioning to reduce the stress on the other relatively rigid structures comprising a SOFC. Simpkins, 5:26-28. In this manner, Simpkins describes structures for increasing the flexibility and durability of SOFCs. Simpkins, 1:59-60. Simpkins also describes expander structures having "a geometry capable of allowing for the expansion and contraction of the fuel cell during operation, thereby providing for a more durable and flexible fuel cell." Simpkins, 6:15-18.

Simpkins also discloses a list of materials that may form interconnects, including "ferrous materials" and "alloys." Simpkins, 6:46-67. However, it is clear that Simpkins focuses almost exclusively on the mechanical and physical structure of SOFCs.

Taruya is relied on by the Examiner as a secondary reference, which the Examiner uses to modify the teachings disclosed in Simpkins. Taruya discloses a solid polymer type fuel cell comprising a ferritic stainless steel separator. The ferritic stainless steel may comprise, among other elements, 10.5-35 weight percent chromium, 0-6 weight percent Mo, less than 0.018 weight percent carbon, less than 0.2 weight percent titanium, and less than 0.3 weight percent niobium. Taruya, para. [0035]-[0049].

Wood discloses a separator plate purportedly suitable for use in various known types of fuel cells. Wood, 1:7-8. Wood is apparently relied on by the Examiner as an ancillary reference, which the Examiner uses as evidence that a person having ordinary

skill in the art would allegedly know that interconnects are interchangeable between solid oxide fuel cells and solid polymer fuel cells.

Appellant submits that the invention recited in the claims under examination does not result from, and would not have been suggested by, the asserted combination of Simpkins, Taruya, and Woods. Appellant also submits that the record evidence of unexpected results confirms that the claimed invention would not have been obvious. For at least those reasons, the rejections should be withdrawn and the claims allowed.

Taruya explains that the components of SOFCs (including interconnects) are not inter-changeable with the same components from other types of fuel cells because of wide differences in fuel cell operating conditions. For example, paragraphs [0005] and [0006] of Taruya read as follows:

[0005]: Although the aforementioned various types of fuel cells are referred to by the common name of "fuel cell", when taking into account the constituent materials of each cell, they need to be regarded as completely different things. This is because the presence of corrosion of constituent materials due to the electrolyte used, the presence of high temperature oxidation which begins to actualize from around 380°C, the sublimation and redeposition of electrolyte, and the performance demanded by the presence of coagulation, etc., particularly anti-corrosion resistance, is completely different for each fuel cell. In actuality, the materials used are various, ranging from graphite materials, to Ni-clad material, high alloys, and stainless steel.

[0006]: It is completely unthinkable to utilize the materials used in commercialized phosphoric acid fuel cells and molten carbonate fuel cells as the constituent material for polymer electrolyte fuel cells.

See the updated Taruya translation provided in the Evidence Appendix, emphases added by Appellant. The above passage pertains to all other constituent materials of a fuel cell (including the interconnect) because of (due to) the electrolyte. The choice of electrolyte drives the conditions – temperature and corrosion especially – in the fuel cell, which affects these other constituents. Taruya explains that it is "completely unthinkable" to use materials (including interconnects) from other kinds of fuel cells in polymer electrolyte fuel cells. It directly follows that it would have been equally unthinkable to use materials from solid polymer type fuel cells (such as the steel described in Taruya) in SOFCs.

One skilled in the art, after considering Taruya, would not have been motivated to use, and indeed would have been taught not to use, any of the constituent materials of the fuel cell described in Taruya in another type of fuel cell, especially one operating at a much higher temperature, such as a SOFC. In this manner, the operating conditions of different types of fuel cells are so drastically different that one skilled in the art would have recognized that materials of construction are not interchangeable between fuel cell types without significant research and development. Such efforts would have been beyond the application of an ordinary level of skill in the art, and indeed, would have necessarily involved an inventive step over the state of the art pertaining to fuel cells.

The Examiner cites Woods as showing "a separator suitable for use in various known types of fuel cells, such as solid oxide fuel cells and polymer electrolyte fuel cells." In essence, the Examiner attempts to disprove what is taught by Taruya regarding the lack of interchangeability of fuel cell components by citing to Woods. Any conflict between Taruya and Woods on this issue, at best, is evidence of the unsettled nature of the issue. The existence of any such conflict between the cited references is not sufficiently definitive to constitute a teaching, contradictory to Taruya, that "...the interconnect (for a solid polymer fuel cell) of Taruya could have been used the interconnect (solid oxide fuel cell) of Simpkins", as the Examiner contends.

Although Woods' column 1 (to which the Examiner cites) does refer to some use of ferrous metal separator plates, it also refers to disadvantages of using ferrous metals in molten carbonate fuel cells:

In molten carbonate fuel cells, in particular, under fuel cell operating conditions, in the range of about 500°C to about 700°C, molten carbonate electrolytes are very corrosive to ferrous metals which, due to their strength, are required for fuel cell housings and separator plates. The high temperature operation of stacks of molten carbonate fuel cells increases both the corrosion and thermal stress problems in the wet seal area, especially when the thermal coefficients of expansion of adjacent materials are different.

Woods, 1:39-48.

A reference must be considered in its entirety, *i.e.*, as a whole, including portions that would lead away from the claimed invention. MPEP § 2141.02(VI); W.L. Gore &

Associates, Inc. v. Garlock, Inc., 721 F.2d 1540 [220 USPQ 303] (Fed. Cir. 1983), cert. denied, 469 U.S. 851 (1984). A reference is said to "teach away" from a claimed invention when it "suggests that the line of development flowing from the reference's disclosure is unlikely to be productive of the result sought by the applicant." In re Gurley, 27 F.3d 551, 553 [31 USPQ2d 1130] (Fed. Cir. 1994). Furthermore, "[a] reference may be said to teach away when a person of ordinary skill, upon reading the reference, would be...led in a direction divergent from the path that was taken by the applicant." Id.; see also Para-Ordnance Mfg. v. SGS Importers Int'l, 73 F.3d 1085, 1090 [37 USPQ2d 1237] (Fed. Cir. 1995).

Insofar as Woods allegedly generally teaches that the disclosed fuel cell separator plate may be used in different types of fuel cells (1:7-10), the reference specifically teaches that ferrous materials would suffer critical problems under operating conditions in the range of about 500-700°C (1:39-48). Accordingly, on balance, Woods would have suggested that ferrous materials are particularly unsuitable for fuels cells operating at greater than 700°C such as SOFCs. In this manner, Woods in essence teaches away from using ferrous materials in SOFCs, and actually reinforces the explicit teaching in Taruya that materials of construction for different types of fuel cells are not generally interchangeable.

Moreover, Appellant submits that the separator plate having an insert ring step design disclosed in Woods, is not necessarily a similar structure as the interconnect disclosed in Simpkins, and therefore, a person skilled in the art would not consider Woods as suggesting interchangeability. The separator plate described in Woods addresses various issues related to wet seals in fuel cells. Woods, 5:16-23. The separator plate described in Woods is illustrated, for example, in Figure 2.

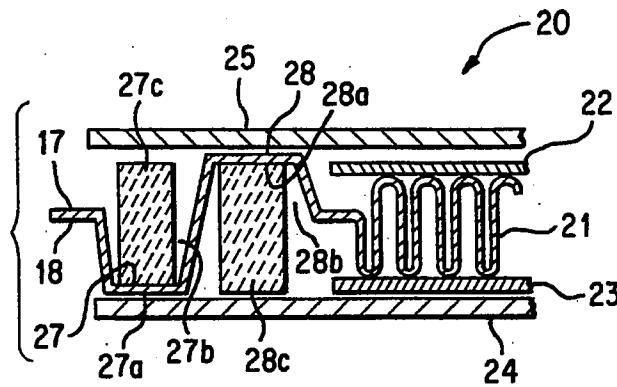
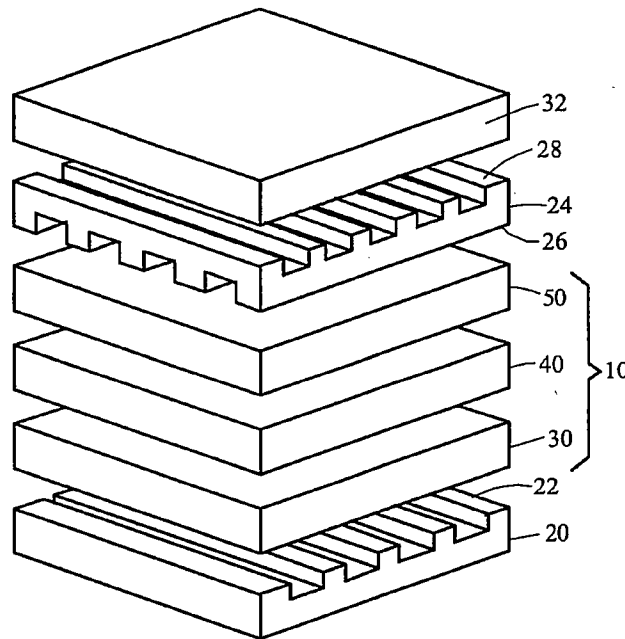


FIG. 2

Woods' separator plate 21 separates electrodes 22 and 23, disposed between electrolytes 24 and 25. As illustrated in Figure 2, for example, the separator 21 is a folded sheet structure. In contrast, the interconnect 24 described in Simpkins, and illustrated in Figure 1 therein, is a monolithic structure.

FIG. 1



A person skilled in the art faced with these two references would not consider the structure described in Woods as suggesting a material interchangeability with the structure described in Simpkins given their apparently different functions.

Furthermore, a person having ordinary skill in the art faced with the teachings of Simpkin and Taruya, and contradictory teachings derived from Woods, would not have been motivated to utilize the material of Taruya in the SOFC of Simpkins with any expectation that the interchange of materials would be successful. For at least this reason, the asserted combination of references fails to teach or suggest the invention recited in claim 36 of the Subject Application.

The Examiner contends that Simpkins discloses that the interconnect described therein may be formed from a ferritic stainless steel. Appellant does not agree with the Examiner. Referring to Figure 2, Simpkins discloses:

The interconnects **80, 82** can be fabricated by tape casting techniques. The thickness of the flat plate structures is about 500 microns or less. The interconnects **80, 82** are preferably stable and electrically conductive at about 800° C. in a dual
50 environment, i.e., an oxidizing atmosphere on one side and a reducing atmosphere on the other side. Some possible interconnects **80, 82** can comprise materials such as silver, copper, ferrous materials, strontium, aluminum, lanthanum, chromium, chrome, gold, platinum, palladium, nickel,
55 titanium, conducting ceramics (e.g., doped rare earth oxides of chromium, manganese, cobalt, nickel, and the like; doped zirconia, including, zirconia doped with titanium, copper, and the like), and the like, as well as alloys, oxides, cermets, composites, and combinations comprising at least one of the
60 foregoing materials. Preferably, the interconnects **80, 82** comprise lanthanum chromite doped with an alkaline earth element. More preferably, the interconnects **80, 82** comprise strontium-doped lanthanum chromite (LSC). Also, metals, such as ferritic stainless steels, nickel, chrome, aluminum
65 alloys, may be coated with LSC or strontium-doped lanthanum manganite (LSM) to achieve the same desired properties.

Simpkins, 6:46-67. According to Simpkins, the interconnects have desired properties including stability and electrical conductivity at about 800°C in oxidizing and reducing atmospheres. The undifferentiated list of materials includes ferrous materials, but goes on to state that ferritic stainless steels may be coated with LSC or LSM to achieve the same desired properties as LSC or LSM interconnects. In this regard, Simpkins suggests that it would be necessary to coat a ferritic stainless steel interconnect to achieve the desired properties for interconnect application.

In contrast, the interconnect recited in claim 36 does not require coating with a ceramic to achieve excellent performance in interconnect applications. Indeed, the particular chemistry of the ferritic stainless steel recited in claim 36 imparts performance enhancements that are not taught or suggested by the prior art.

The Examiner contends that Taruya suggests the compositional features of the ferritic stainless steel recited in claim 36, and that the recited CTE and creep properties would have been inherent. Appellant does not agree with the Examiner. Comparing the teachings of Taruya and the steel composition recited in claim 36 reveals the following:

Element	Taruya	Claim 36
Cr	10.5-35	> 25
Mo	0-6	0.75-1.5
C	< 0.018	≤ 0.05
Nb	< 0.3	$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$
Ti	< 0.2	$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$
Ta	0	$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$

Molybdenum may impart conflicting properties to a ferritic stainless steel. Molybdenum tends to reduce the CTE and provides solid solution strengthening. In conjunction with niobium, molybdenum will form a strengthening Laves phase $Fe_2(Nb,Mo)$ precipitate. However, molybdenum also substantially increases the tendency of stainless steel to precipitate undesirable sigma phase as well as chi phase (Fe,Cr,Mo). A relatively high level of molybdenum also impairs the oxidation resistance of steel and can, under certain circumstances, promote a catastrophic form of oxidation. For these reasons, the molybdenum content of the steel recited in claim 36 is carefully controlled within 0.75-1.5 weight percent. Specification, page 13, lines 8-14.

In contrast, Taruya discloses an extremely broad 0-6 weight percent range of molybdenum. This wide range and potentially very high level of molybdenum could result in a stainless steel having unacceptable levels of undesirable sigma phase and chi phase precipitates. Moreover, the high levels of molybdenum possible in the Taruya

alloy could result in an unacceptably low CTE. The fact that Taruya emphasizes large alloying additions of molybdenum clearly indicates a lack of appreciation and understanding in the prior art concerning the chemistry of ferritic stainless steels, particularly as it relates to SOFC interconnect applications.

Regarding the equation $0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$, Appellant submits that Taruya fails to teach or suggest the criticality of this relationship among carbide forming elements, and its implications. First, Taruya teaches less than 0.3 weight percent niobium, less than 0.2 weight percent titanium, and zero tantalum. Accordingly, it would be impossible for the Taruya alloy to ever satisfy the equation recited in claim 36.

The Examiner contends that the CTE and creep properties recited in claim 36 would have been inherent in the Taruya alloy. Appellant does not agree with the Examiner.

“To establish inherency, the extrinsic evidence must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill. Inherency, however, may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.” MPEP § 2112.IV (citations omitted). “In relying upon the theory of inherency, the examiner must provide a basis in fact and/or technical reasoning to reasonably support the determination that the allegedly inherent characteristic necessarily flows from the teachings of the applied prior art.” MPEP § 2112.IV (citations omitted) (emphasis in original).

The Examiner cannot just deem that the material properties would have been inherent in the Taruya alloy given the compositional distinctions described above. This amounts to mere speculation and conjecture. The extrinsic evidence (i.e., the technical disclosure in Taruya) must make clear that the alleged inherent properties would necessarily be present in the Taruya alloy. Here, the Examiner is relying upon a theory of inherency, but the Examiner never provides a basis in fact and/or technical reasoning to reasonably support the determination that the allegedly inherent properties

necessarily flow from the teachings of Taruya. See MPEP § 2112.IV. As a result, the Examiner's inherency argument is improper.

Appellant submits that any claim not expressly discussed in this Appeal Brief is patentable for at least the reasons discussed above because such claims depend from the independent claim discussed above, and therefore, such claims incorporate by reference all of the patentable features discussed above.

As discussed above, Simpkins and Taruya, alone or in combination, or when considered in light of Woods, fail to teach or suggest a SOFC as recited in claim 36. A person skilled in the art considering the cited references at the time of the present invention would not have found the features recited in the present claims to be obvious or inherent. Therefore, Appellant respectfully submits that a *prima facie* case under § 103(a) has not been established by the Examiner. Accordingly, Appellant respectfully requests withdrawal of the § 103(a) rejections.

(B) IF A *PRIMA FACIE* CASE UNDER 35 U.S.C. § 103(a) HAS BEEN ESTABLISHED, THE RECORD EVIDENCE OF UNEXPECTED RESULTS NEVERTHELESS REBUTS ANY *PRIMA FACIE* CASE.

As described above in Section (b), Taruya does not teach or suggest an alloy satisfying the equation:

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$$

(hereinafter the "Recited Range"). Taruya teaches an alloy composition including less than 0.2% Ti, and less than 0.3% Nb. Taruya does not mention any concentration of Ta whatsoever. Thus, the total combined concentration of Nb, Ti, and Ta taught in Taruya is less than 0.5%, which clearly is outside the Recited Range. Accordingly, Taruya does not teach an alloy composition as recited in the claims of the Subject Application.

Nevertheless, the Examiner alleges that "the courts have ruled a *prima facie* case of obviousness exists where the claimed ranges and prior art ranges do not

overlap but are close enough that one skilled in the art would have expected them to have the same properties,” and that “Applicant has not shown the ferritic stainless steel alloy of Taruya necessarily has different properties than the ferritic stainless steel alloy of the claimed invention.”

MPEP § 2144.05 states that “Applicants can rebut a *prima facie* case of obviousness based on overlapping ranges by showing the criticality of the claimed range...In such a situation, the applicant must show that the particular range is critical, generally by showing that the claimed range achieves unexpected results relative to the prior art range.” As discussed below, the Recited Range of claim 36 results in alloy compositions having properties that were unexpected and surprising relative to compositions including a combined concentration of Nb, Ti, and Ta that is outside the Recited Range.

As discussed in the specification of the Subject Application, six heats of ferritic stainless steel were prepared and tested for various physical properties. The compositions of the heats are shown in the following table, which is reproduced from page 21 of the specification:

Heat	WC70	WC71	WC72	WC73	WC74	WC75
C	0.0026	0.0026	0.0038	0.0022	0.0023	0.0033
Mn	0.054	0.055	0.060	0.049	0.052	0.053
P	0.010	0.010	0.010	0.010	0.010	0.010
S	0.0029	0.0027	0.0014	0.0011	0.0003	0.0006
Si	0.16	0.15	0.14	0.15	0.15	0.15
Cr	25.52	25.98	25.63	25.77	25.69	25.79
Ni	0.096	0.094	0.095	0.094	0.094	0.095
Mo	1.05	1.05	1.03	1.04	1.04	1.04
Al	0.002	0.002	0.002	0.002	0.002	0.002
Nb	0.12	0.68	0.13	0.68	0.71	0.71
Ce	<0.001	<0.001	0.001	0.003	0.042	0.009
La	<0.001	<0.001	0.001	0.001	0.016	0.003
Zr	<0.001	<0.001	<0.001	<0.001	<0.001	0.011
N	0.0010	0.0010	0.0008	0.0009	0.0011	0.0011

Of the three elements included in the Recited Range, Nb was included in each of the above heats. Heats WC70 and WC72 are representative of a conventional ferritic stainless steel and do not satisfy the Recited Range. The remaining heats included

higher levels of Nb and their compositions fall squarely within claim 1, including satisfying the Recited Range. As discussed in the specification, ferritic stainless steels may provide a good thermal expansion match with the ceramic electrolytes of SOFCs, but such steels typically exhibit low creep resistance, which is detrimental to their use in SOFCs.

The present inventor unexpectedly discovered that relatively small increases in Nb concentration in certain ferritic stainless steel compositions yield a significant and surprising improvement in creep resistance at SOFC operating temperatures. The increased Nb concentration also surprisingly resulted in significant improvements in microstructural stability and certain other mechanical properties. Given the inventor's understanding of the mechanism that may be responsible for the unexpected and substantial improvement in alloy properties resulting from increases in Nb concentration, the inventor further concluded that Ti and/or Ta may be included in the alloy, either along with or in place of Nb, in a combined concentration that falls within the Recited Range to achieve similarly significant improvements in the properties.

Pages 24-26 of the Subject Application's specification as originally filed discuss the improvements in microstructural stability of the experimental heats included in the above table. The alloy compositions included in the table that do not satisfy the Recited Range readily recrystallized at 1750°F and also experienced significant grain growth at temperatures of about 1950°F and above. In contrast, the alloys listed in the above table that do satisfy the Recited Range did not show any evidence whatsoever of recrystallization until heated to the substantially higher temperature of about 2000°F. Therefore, providing an alloy composition within the Recited Range unexpectedly and surprisingly inhibited recrystallization, preventing the phenomenon until the alloy was heated to temperatures substantially greater than the recrystallization temperature of the tested conventional alloys.

Similarly, pages 27-28 of the specification as originally filed describe the unexpected and significant improvements in yield strength, tensile strength and hardness exhibited by the alloys listed in the above table that satisfy the Recited Range.

Creep strength is discussed on pages 28-39 of the specification the Subject Application. The creep strength of conventional ferritic stainless steel alloys was considered inadequate at the temperatures and under the mechanical stresses to which interconnects are subjected in SOFC applications. Modification of alloy compositions to satisfy the Recited Range, however, was unexpectedly found to significantly improve creep resistance. The alloys listed in the above table satisfying the Recited Range surprisingly exhibited significantly superior creep resistance compared to the conventional alloys, and the improvement was especially significant at higher test temperatures. For example, alloys satisfying the Recited Range exhibited creep rupture strength of 400 hours at 1000 psi and 900°C. In contrast, the conventional alloys exhibited creep rupture strength of only 156 hours at lower pressure (900 psi) and lower temperature (900°C). This is summarized in the following table:

	Creep Rupture Strength (psi)	Temperature (°C)	Time (hrs)
Alloys within the Recited Range	1000	900	400
Alloys below the Recited Range	900	900	156

The alloys listed in the above table having compositions satisfying the Recited Range exhibited a time to 1% creep strain of at least 100 hours at 900°C under a load of 1000 psi. In contrast, the conventional alloys evaluated exhibited a time to 1% creep strain of only 2.5 hours at 900°C under a load of 900 psi. This is summarized in the following table:

	Time to 1% Creep Strain (hrs)	Temperature (°C)	Load (psi)
Alloys within the Recited Range	> 100	900	1000
Alloys below the Recited Range	2.5	900	900

Also, the tested alloys satisfying the Recited Range exhibited a time to 2% creep strain of at least 200 hours at 900°C under load of 1000 psi. In contrast, the

conventional alloys evaluated exhibited a time to 2% creep strain of only 5 hours at 900°C under load of 900 psi. These results are summarized in the table, below.

	Time to 2% Creep Strain (hrs)	Temperature (°C)	Load (psi)
Alloys within the Recited Range	> 200	900	1000
Alloys below the Recited Range	5	900	900

Therefore, as clearly demonstrated in the specification of the Subject Application, adjusting the composition of a ferritic stainless steel alloy to satisfy the Recited Range (as well as to meet the other limitations recited in claim 36) results in a very significant and unexpected improvement in several properties including microstructural stability, yield strength, tensile strength, hardness, and perhaps most significantly, creep strength and time to 1% and 2% creep strain.

These significant improvements yield a ferritic stainless steel suitable for use as interconnect materials in SOFCs, an application which prior to the present invention required use of substantially more expensive ceramic materials. Thus, it has been shown that an alloy as recited in claim 36 is novel and would have been non-obvious for at least the reason that it satisfies the Recited Range. The Subject Application also clearly shows that alloy compositions satisfying the Recited Range exhibit unexpected, surprising, and significant improvement in several properties, including properties critical to materials used in SOFC interconnect applications. That the Recited Range results in these unexpected, surprising, and significant improvements clearly rebuts any *prima facie* case of obviousness established by the cited combination of references.

Therefore, adjusting the composition of a ferritic stainless steel alloy to satisfy the Recited Range provided a significant and unexpected improvement in several properties including creep properties, microstructural stability, yield strength, tensile strength, and hardness. These significant improvements yield a ferritic stainless steel suitable for use as interconnect materials in SOFCs, which provides many advantages over conventional ceramic interconnect materials.

VIII. CLAIMS APPENDIX

36. A solid oxide fuel cell comprising:

an anode;

a cathode;

an electrolyte comprising stabilized zirconia, wherein the electrolyte is intermediate the anode and the cathode; and

an interconnect providing a current pathway from the anode, the interconnect comprising a ferritic stainless steel comprising:

greater than 25 weight percent chromium,

0.75 up to 1.5 weight percent molybdenum,

up to 0.05 weight percent carbon, and

at least one of niobium, titanium, or tantalum, wherein the weight percentages of niobium, titanium, and tantalum satisfy the equation

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1,$$

wherein the steel has a coefficient of thermal expansion within about 25 percent of the coefficient of thermal expansion of stabilized zirconia between 20°C and 1000°C and exhibits at least one creep property selected from the group consisting of creep rupture strength of at least 1000 psi at 900°C, time to 1% creep strain of at least 100 hours at 900°C under load of 1000 psi, and time to 2% creep strain of at least 200 hours at 900°C under load of 1000 psi.

37. The solid oxide fuel cell of claim 36, wherein the coefficient of thermal expansion of the steel is at least as great as the coefficient of thermal expansion of stabilized zirconia between 20°C and 1000°C.

38. The solid oxide fuel cell of claim 36, wherein the coefficient of thermal expansion of the steel is at least as great as the coefficient of thermal expansion of yttria-stabilized zirconia between 20°C and 1000°C.

39. The solid oxide fuel cell of claim 36, wherein the steel comprises:

25 up to 35 weight percent chromium;

0.75 to less than 1.5 weight percent molybdenum;

up to 0.005 weight percent carbon; and

at least one of niobium, titanium, or tantalum, wherein the steel includes no more than 0.50 weight percent titanium, and the weight percentages of niobium, titanium, and tantalum satisfy the equation

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 0.75.$$

40. The solid oxide fuel cell of claim 36, wherein the steel includes no more than 0.50 weight percent titanium.

42. The solid oxide fuel cell of claim 36, wherein the weight percentages of niobium, titanium and tantalum satisfy the equation

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 0.75.$$

43. The solid oxide fuel cell of claim 36, wherein the steel includes no more than 0.50 weight percent titanium and the weight percentages of niobium, titanium, and tantalum satisfy the equation

$$0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1.$$

IX. EVIDENCE APPENDIX

The content of this Evidence Appendix includes the following:

- English language translation of Japanese Patent Application Publication No. 2000-294256 to Tarutani et al. (referred to throughout the prosecution of the Subject Application as "Taruya"), Certificate of Translation dated May 23, 2005.
- Review/Edit of English language translation of Japanese Patent Application Publication No. 2000-294256, Certificate of Translation dated May 25, 2007.



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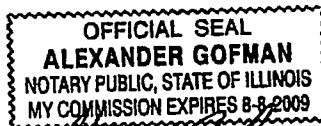
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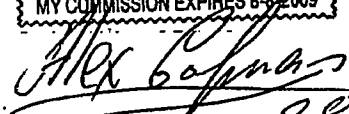
I, Dwight Sora, hereby certify that I am competent in both English and Japanese languages. I further certify that under penalty of perjury translation of the aforementioned patent

Patent H2000-294256

from the Japanese language into the English language is accurate and correct to the best of my knowledge and proficiency.


Professional Translator




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Claim for Examination Yes No. of Claims 4 OL (Total 10 Pages)

(21) Patent Application	Patent H11-103357	(71) Applicant	000002118 Sumitomo Metal Industries, Ltd. 5-33, Kitahama 4-Chome, Chuo Ward, Osaka City, Osaka Prefecture
(22) Application No.	April 9, Heisei 11 (4/9/1999)	(72) Inventor	Yoshio Tarutani Sumitomo Metal Industries, Ltd. 5-33, Kitahama 4-Chome, Chuo Ward, Osaka City, Osaka Prefecture
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			Continued on last page

(54) [Title of Invention] Solid Polymer-Type Fuel Cell

(57)[Abstract of the Disclosure]

[Problem] To provide a solid polymer-type fuel cell equipped with a stainless steel separator in which the poison of each electrode-supported catalyst due to eluted metal ions is low.

[Means to Solve Problem] A solid polymer-type fuel cell equipped with a separator composed of stainless steel in which the impurities of S, P, V, Ni and CU, etc. have been lowered, and the Cr and Mo content is made to satisfy Cr: 10.5~35% and Mo: 0.5~5%, moreover, $12\% \leq \text{Cr} + 3\text{Mo} \leq 54\%$.

[Scope of Claims]

[Claim 1] A solid polymer-type fuel cell characterized by a separator being composed of ferrite stainless steel that has the chemical composition mentioned below, with a solid polymer-type fuel cell that is made to generate direct current by supplying fuel gas and oxidizing gas, on a

It contains, by weight percent,
 S: Less than 0.005%,
 V: Less than 0.2%,
 CU: Less than 0.2%,
 Mo: 0~6%,

and (Cr + 3Mo) is within a range of 10.5~43%

[Claim 2] A solid polymer-type fuel cell as in Claim 1 characterized by the Si and Mn in the ferrite stainless steel being, by weight percent, less than 0.3% for Si weight and less than 0.4% for Mn.

[Claim 3] A solid polymer-type fuel cell as in Claim 1 or 2 for which the C and N weight in the ferrite stainless steel is, by weight percent, C: less than 0.018%, N: less than 0.018%, and the total C and N content being less than 0.025%.

[Claim 4] A solid polymer-type fuel cell as in any of the Claims 1 thru 3 for which the ferrite stainless steel contains one or two types of Ti and Nb, Ti is less than 0.2% by weight percent and within a range of 6 (C% + N%)~25 (C% + N%), and Nb is less than 0.3% and within a range of 6C%~25C%.

[Detailed Description of Invention]

[0001]

[Field of industrial Application] This invention is related to solid polymer-type fuel cells in which a small distributed generator, such as one meant for automobile installation or one meant for domestic use, is used.

[0002]

[Prior Art] A fuel cell is a battery that generates direct current power by using hydrogen and oxygen, of which there are solid electrolyte-type fuel cells, fused carbonate-type fuel cell, phosphoric acid fuel cell, and solid polymer-type fuel cells. The name of a fuel cell is derived from the constituent material of the "electrolyte" part that basically makes up the battery.

[0003] At present, with regards to fuel cells achieving the commercial level, there are phosphoric acid-type fuel cells and fused carbonate-type fuel cells. The rough operating temperatures for fuel cells are 1000°C for solid electrolyte-type fuel cells, 650°C for fused carbonate-type fuel cells, 200°C for phosphoric acid-type fuel cells, and 80°C for solid polymer-type fuel cells.

[0004] It is easy to start and stop solid polymer-type fuel cells when the operating temperature is low at 80°C before and after, and because it is possible for the energy efficiency to be about 40%, there is anticipation for its global implementation as an emergency distributed generator for small offices and telephone exchanges, a domestic small distributed generator that assumes city gas as fuel, and a power source for installation in low-emission electric automobiles that assume hydrogen gas, methanol or gasoline as fuel.

[0005] With each of the aforementioned types of fuel cells, in cases when we think about the individual constituent

laminated body that has centered a solid polymer electrolyte membrane and layered unit batteries that have superposed fuel electrode membranes and oxide electrode membranes by placing a separator between several and unit batteries.

P: Less than 0.025%,
 Ni: Less than 0.2%,
 Cr: 10.5~35%,
 Rare Earth Metals: 0~0.1%

materials of items that are referred to by the common name of "fuel cell," it is necessary for them to be batched as completely different things. This is because the performance obtained by the absence of constituent materials corroding due to the electrolytes used, the absence of high-temperature oxidation that starts to actualize from around 380°C, and the absence of sublimation, reprecipitation, and coagulation of electrolytes, especially corrosion-resistance performance, is completely different for each fuel cell. Actually, the materials used are many, varying from graphite material to Ni clad material, high alloy and stainless steel.

[0006] It is not at all possible to consider the application of material used in commercialized phosphoric acid-type fuel cells and fused carbonate-type fuel cells in the constituent material of solid polymer-type fuel cells.

[0007] Fig. 1 is a drawing showing the construction of a solid polymer-type fuel cell, Fig. 1(a) is an exploded view of a fuel cell (single cell), Fig. 1 (b) is an oblique drawing of an entire fuel cell. As shown in the same drawing, the fuel cell 1 is an aggregate of single cells. A single cell is constructed, as shown in Fig. 1 (a), with fuel electrode membranes (anode) 3 layered on one face of a solid polymer electrolyte membrane 2, and oxide electrode membranes (cathode) 4 layered on the opposite face, with separators 5a and 5b superimposed on both faces.

[0008] As for a representative solid polymer electrolyte membrane 2, there is a fluorine ion exchange resin layer that has a hydrogen ion (proton) exchange group.

[0009] A catalytic layer composed of fluorine resin that has particulate platinum catalysts, graphite powder, and, as needed, a hydrogen ion exchange group, is provided on a fuel electrode membrane 3 and an oxide electrode membrane 4, and making contact with the fuel gas or oxidative gas.

[0010] Hydrogen is supplied to a fuel electrode membrane 3 by fuel gas (hydrogen or oxygen-containing gas) A being injected from a channel 6 provided on a separator 5a. In addition, from a channel 6b provided on a separator 5b, oxidative gas B such as air is injected, and oxygen is supplied. Direct current power is generated by an electrochemical reaction produced by the supply of these gases.

[0011] Anode Side: $H_2 \rightarrow 2H^+ + 2e^-$ Cathode Side: $(1/2) O_2 + 2H + 2e^- \rightarrow H_2O$ (3)

The functions obtained by the solid polymer-type fuel cell separator are (1) function as a "channel" that uniformly supplies fuel gas in-plane on the fuel electrode side, (2)

function as a "channel" that efficiently discharges water generated on the cathode side, in addition to post-reaction air and oxygen carrier gas from the fuel cell, to outside of the system, (3) function as an electrical "connector" between the individual cells that maintains low electrical resistance and good electrical conductivity as an electrode over a long period of time, and (4) function in adjoining cells as a cathode chamber for the cell and a "partition" adjacent to the anode chamber for the cell on other side.

[0012] Up to now, the use of cathode sheet material as a separator material has come to be keenly investigated, but there has been a problem with it being "easy to fracture" the cathode sheet material, and furthermore, there have been problems with the machining cost for smoothing the surface and the machining cost for forming the gas channel becoming quite expensive. These are each unavoidable problems, and there are conditions in which they inevitably make commercialization of fuel cells difficult.

[0013] The most attention is being paid to thermally expandable graphite finished goods as material for solid polymer-type fuel cell separators because of the considerable low cost. However, in order to confer function as the aforementioned partition to reduce gas permeability, resin impregnation and baking that reaches "multiple times" must be implemented. In addition, issues to resolve in future, such as the machining costs for insuring flatness and groove formation, are many, and, at present, are not at the point that they will be realized.

[0014] With regards to activities for confronting the investigation into application of these graphite materials, attempts to apply stainless steel to the separator for the purposes of cost reduction are being started.

[0015] In the bulletin for Patent H10-228914, an announcement is made of fuel cell separators that are composed of metal material and employ gold plating directly to the face that makes contact with the electrode of the unit battery. Stainless steel, aluminum and nickel-iron alloy are provided as metal materials, while SUS304 is being used for stainless steel. In this invention, since gold plating is being employed with the separator, the contact resistance between the separator and electrode is reduced, and conduction of electrons from the separator is becomes good, so it assumed that the output voltage of the fuel cell is enlarged.

[0016] In the bulletin for Patent H08-180883, an announcement is made of solid polymer electrolyte-type fuel cells that use separators composed of a metal material on which the passivated film that forms on the surface is easily produced by air. Stainless steel and titanium alloy are provided as metal materials. In this invention, passivated film always exists on the surface of the metal used in the separator, and the amount of ionization of the water generated by an individual fuel cell by the surface

(2) A solid polymer-type fuel cell as in the aforementioned (1) in which the Si and Mn in the ferrite stainless steel is, by weight percent, less than 0.3% for Si weight and less than 0.4% for Mn, and which contains, by weight percent,

S: Less than 0.005%,
V: Less than 0.2%
CU: Less than 0.2%
Mo: 0~6%

of the metal becoming chemically difficult to violate is reduced, and it is assumed that drops in the electrochemical reactivity of an individual fuel cell are inhibited.

[0017] However, even if metal material such as the stainless steel that provides the passivated film on the surface as announced in the aforementioned public bulletin is used as is on the separator, elution of metal occurs without sufficient corrosion resistance, the performance of the supported catalyst deteriorates (henceforth described as supported catalyst poison) due to eluted metal ions. In addition, because of problems with the contact resistance of the separator increased by corrosion product such as Cr-OH and Fe-OH produced after elution, at present, noble metal plating such as gold plating in which cost is disregarded is applied to separators composed of metal materials.

[0018] With regards to its use with separators for metal materials up to now, there have been results in which it has been applied, but they are at a stage far from practical implementation.

[0019] It is possible to use them as a separator with "purity" that will not have high-cost surface treatment performed, and if it is possible to develop stainless-steel with excellent corrosion resistance, in addition to excellent electrical conductivity in battery environments, the manufacturing cost of fuel cells will greatly drop, and it will be possible to expect commercialization and increased use of solid polymer-type fuel cells.

[0020]

[Problems to be solved by Invention] The problem for this invention is to provide a solid electrolyte-type fuel cell equipped with a stainless steel separator in which the increase in contact electrical resistance with the electrode due to corrosion product and the increase in contact resistance due to reinforcement of the passivated film is small, without performing high-cost surface treatment, and in which the poison of each electrode-supported catalyst due to eluted metal ions is quite low.

[0021]

[Means to solve Problem] A summary of this invention is as follows below.

(1) A solid polymer-type fuel cell characterized by a separator being composed of ferrite stainless steel that has the chemical composition mentioned below, with a solid polymer-type fuel cell that is made to generate direct current by supplying fuel gas and oxidizing gas, on a laminated body that has centered a solid polymer electrolyte membrane and layered unit batteries that have superposed fuel electrode membranes and oxide electrode membranes by placing a separator between several and unit batteries.

[0023]

(4) P: Less than 0.025%
Ni: Less than 0.2%
Cr: 10.5~35%
Rare Earth Metals: 0~0.1%

and (Cr+3Mo) is within a range of 10.5~43%.

[0024] A solid polymer-type fuel cell as in the aforementioned (1) or (2) for which the C and N weight in the ferrite stainless steel is, by weight percent, C: less than 0.018%, N: less than 0.018%, and the total C and N content being less than 0.025%.

[0025] (4) A solid polymer-type fuel cell as in any of the above entries 1 thru 3 for which the ferrite stainless steel contains one or two types of Ti and Nb, Ti is less than 0.2% by weight percent and within a range of 6(C%+N%)~25(C%+N%), and Nb is less than 0.3% and within a range of 6C%~25C%.

[0026] Furthermore, the separator mentioned is one that has the four functions described previously. Specifically, (a) function as a "channel" that uniformly supplies fuel gas in-plane on the fuel electrode side, (b) function as a "channel" that efficiently discharges water generated on the cathode side, in addition to post-reaction air and oxygen carrier gas from the fuel cell, to outside of the system, (c) function as an electrical "connector" between the individual cells that maintains low electrical resistance and good electrical conductivity as an electrode over a long period of time, and (d) function in adjoining cells as a cathode chamber for the cell and a "partition" adjacent to the anode chamber for the cell on other side. There are also cases of a construct to which these functions are assigned by multiple sheet layers. The separator referred to in this invention is a sheet that has at least one of the functions from among the four mentioned above.

[0027] In order to develop a solid polymer-type fuel cell equipped with a separator composed of stainless steel, the inventors implemented various tests by using single cells in the environments in which the separator is placed, for the purpose of reducing the metal ions eluted from the stainless steel surface as much as possible. According to the results, in 1~3 pH environments in which a) separators that come to complete this invention by achieving the following findings are placed (henceforth referred to simply as separator environments), the corrosion resistance of austenitic stainless steel is insufficient, and inapplicable to separators with significant elution of metal.

[0028] In separator environments b), ferrite stainless steel demonstrates good corrosion resistance, and in general ferrite stainless steel, metal elution of a degree that will effect battery performance will be produced.

[0029] When c) metal is eluted, corrosion product (hydroxide with Fe assumed to be the subject) is generated and an increase in contact electrical resistance is brought about, causing significantly negative effects on the performance of the supported catalyst, so the battery performance represented by electromotive force deteriorates over a short time. In addition, it causes a negative effect on the positive ion conductivity of fluorine ion exchange resin layer that has a hydrogen ion (proton) exchange group.

[0030] d) In order to prevent elution of metal, the passivated film must be rigidified within the impurities inside of ferrite stainless steel, in addition to simultaneously reducing the S, P, V, Ni and Cu content.

[0031] e) Even when the passivated film is rigidified, the contact electrical resistance will intensify when the passivated film thickness is thickened, and the battery efficiency will significantly drop.

[0032] f) In order to control elution of metal in separator environments by rigidifying passivated film without thickening, it is necessary for the Cr and Mo content to be within a range of 12~43%.

[0033] g) By actively adding Mo, the corrosion resistance is insured, but even if it is assumed that the Mo has eluted, the effect on the performance of catalysts supported by anode and cathode parts is comparatively slight.

[0034]

[Mode for Invention] Below, we will give a detail explanation of the reasons for defining the chemical composition of a separator composed of ferrite stainless steel provided by a solid polymer-type fuel cell in this invention. Furthermore, the % expression of the aforementioned elements indicates weight percent.

[0035] S: intra steel S weight must be assumed to be less than 0.005%. S, corresponding to the coexisting intra steel elements and intra steel S weight, is almost completely precipitated as complex nonmetallic inclusions like Mn-sulfide, Cr-sulfide, Fe-sulfide, Ti-sulfide, and complex sulfides and oxides of these. However, in separator environments, nonmetallic inclusions of any composition also act as a source of corrosion for ones that have differences in degree, and are harmful to maintenance of passivation and control of corrosion and elution of metal.

[0036] In addition, under the conditions in which fuel cells are operated, the pH inside of the gap between separators composed of ferrite stainless steel and the MEA (Membrane Electrode Assembly) drops due to occurrence of cell reaction and/or oxygen concentration differential cell corrosion, and although conditions will arise in which micro cell corrosion easily occurs, on such occasions the nonmetallic sulfide inclusions will have large effects as sources of corrosion and acceleration factors. The intra steel S weight of regular mass-produced steel exceeds 0.005% at around 0.008%, but it must be reduced to less than 0.005% in order for the aforementioned detrimental effects to be prevented. The desirable intra steel S weight is less than 0.002%, while the most desirable intra steel S weight level is less than 0.001%, and if low, then a low level is acceptable.

[0037] P: intra steel P weight must be less than 0.025%. The content level of regular commercial stainless steel is about 0.026~0.035%. P is the inevitable impurities, and, while ranked with S, is a hazardous element that has little effect on the poisons in the anode and cathode catalytic layers. A low level is desirable.

[0038] V: intra steel V must be set to less than 0.2%. In general, V is contained as an impurity inside of the Cr source that is the essential raw dissolution material when ingoting stainless steel, while a mixture at some level is inevitable. However, the eluted V has little negative effect on the performance of catalysts supported by the anode and cathode parts. In addition to maintaining

battery characteristics, the tolerable upper limit is 0.2%, and if low, then low is acceptable.

[0039] Cu and Ni: intra steel Cu and Ni must be set to less than 0.2% for either one or both. In general, even with small impurity level amounts of Cu and Ni, there are effects of improving the corrosion resistance of stainless steel in low pH environments. However, in separator environments, even with small amounts of Cu and Ni ion elution in passivation maintenance conditions, they lead to an effect on the poisons in anode and cathode catalytic layers, so it is necessary to assume an upper limit of 0.2%. Including a mixture from scrap as well, it is more desirable for the intra steel Cu and Ni weight to be low, but Cu and Ni at an impurity level of the extremely small amount of less than 0.2% raises the passivation of ferrite stainless steel, also has the effect of controlling metal elution in a state of passivation, and, because it improves the battery performance as a result, assumes an upper limit of 0.2%.

[0040] Cr, Mo: Cr and Mo are the basic alloying elements that originally insure corrosion resistance. Corrosion resistance is as high as their content, but is a tendency towards a drop in normal temperature tenacity that accompanies Cr becoming high, and when the Cr weight exceeds 35%, mass production becomes difficult. In addition, at less than 10.5%, even if other elements are changed, it becomes difficult to insure the required corrosion resistance for the separator.

[0041] Mo is an element that is included as required, and in comparison to Cr, has the effect of improving corrosion resistance in small quantities. At less than 0.5%, the effect of Mo becomes unclear. In cases when it is included, when it is included in excess of 6%, it becomes difficult to avoid precipitation of intermetallic compounds such as Sigma phase, and production becomes difficult because of problems with steel embrittlement, so an upper limit of 5% is assumed. By actively including Mo, corrosion resistance is insured. Even when it is assumed that Mo is eluted, the effect on the performance of catalysts supported by anode and cathode parts is comparatively slight, and the negative effect on positive ion conductivity for the fluorine ion exchange resin layer that has a hydrogen ion (proton) exchange group is small.

[0042] (Cr% + 3Mo%): The stainless steel for separators is in a state of passivation in environments of 70°C to a high 100°C, which is the operating temperature for a solid polymer-type fuel cell, and requires that the contact electrical resistance be even successively low. It is necessary to control the increase in passivated film thickness and corrosion product generation within a practical range. As a required condition for this, the Cr and Mo content is the corrosion index (Cr% + 3Mo%), but it is necessary for it to be within a range of 12-43%.

[0043] It is necessary for there to be at least passivation "within 80°C aqueous sulfuric acid solution of 2.6 pH at 25°C" that is determined to be appropriate as a simulated ambient condition of the battery interior in actual operating conditions. For this to be so, it is desirable that the passivation maintenance current density be less than 50 $\mu\text{A}/\text{cm}^2$ at 0.2V vs. SCE in this environment.

[0044] Si: It is desirable that the intra steel Si weight be set to less than 0.3%. Si is as effective a deoxidizing element as Al in mass-produced steel. However, since eluted Si has little effect on the poisons in the anode and cathode catalytic layers, and is clearly harmful with regards to maintaining battery characteristics, less than 0.3% is assumed. Mass production at approximately 0.25% is the most desirable from the viewpoint of reducing mass production costs, but in separator environments, less than 0.2% is desirable. Even more desirable is less than 0.1%.

[0045] Mn: It is desirable that the intra steel Mn weight be assumed to be less than 0.4%. Normally, Mn has a fixing action as a Mn sulfide, and has the effect of improving hot workability. In addition, it is acceptable if it is actively added as a deoxidizing element or a Ni balance compensation element. However, even when in a state in which passivation is being maintained, metal elution barely advances at a time, but when the amount exceeds 0.4%, eluted Mn ions have little effect on the poisons in anode and cathode catalytic layers. Less than 0.1% is desirable. At less than 0.45 Mn, it is possible to manufacture without the generation of problems such as hot cracking during mass production. Hardly any problems with increased manufacturing costs will arise either.

[0046] C, N: Intra steel C and N for the purposes of improving normal temperature tenacity, C is less than 0.018%, N is less than 0.018%, with it desirable if less than 0.025% is assumed at the C%+N% value. C and N are penetrating-type elements, and become a source of deterioration in the base metal tenacity, weld corrosion resistance and tenacity, of high purity ferrite stainless steel. The strict limiting of C and N is a normal temperature tenacity measure, that becomes a problem in manufacturing processes for hot-rolled coils of high purity ferrite stainless steel, but makes it possible avoid rises in manufacturing costs. If intra steel C and N are lowered extremely, normal temperature tenacity will be improved, so if low, then a low level is acceptable.

[0047] Ti: Ti is less than 0.2% as needed, and is included by a range amount greater than six times and less than 25 times the value of (C%+N%). Ti leads to poisons in the anode and cathode catalytic layer, so it is intrinsically an element that must be reduced, but, from the viewpoint of insuring manufacturability during mass production and workability of the plating, the minimum amount is included according to need.

[0048] In cases in which weldability is not demanded, greater than (C% + N%) value x six times and less than 10 times is the most desirable. In cases in which weldability is demanded, greater than 10 times the (C% + N%) value and less than 16 times is the most desirable. In order to avoid the generation of sheet surface defects that are a source of debris, less than 0.1% is desirable. When more than needed is included, it becomes a source of anode and cathode catalytic performance degradation due to elution of metals while in a state in which passivation is maintained.

[0049] Nb: Nb is an element that is included as needed, and is also an alloying element for which the bonding strength with intra steel C and N is stronger than Cr, the same as Ti. Nb is less than 0.3%, and is included within a range of $C\% \times 6 \sim C\% \times 25$ [specifically, $Nb(\%) / C(\%) = 6 \sim 25\%$]. It is quite effective in improving tenacity, including normal temperature tenacity of hot-rolled coils. However, Nb eluted along with corrosion accumulates as a corrosion product on the corrosion face, and has the harmful effect of raising contact electrical resistance, so it is more desirable if the Nb content is low from the viewpoint of base material performance. However, the required minimum amount is added in the cases of insuring weld performance, or determining the necessity of improving the workability of cold-rolled steel sheet material by simultaneously including Nb and Ti.

[0050] Rare Earth Metals (REM): The bonding strength with S at the molten steel stage is quite strong with rare

earth metals, so it has the effect of making S harmless. Accordingly, it is acceptable if it is added in a form like misch metal as needed. Content achieves sufficient effects at less than 0.1%.

[0051] It is acceptable if elements other than the aforementioned elements are included as needed. For example, it is good to include Ca, Mg or B less than 0.1 to improve hot workability.

[0052]

[Working Example] We dissolved ferrite stainless steel of the 28 types of chemical compositions as shown in Table 1 in a high frequency induction heating method 150 kg vacuum melting furnace. We carefully selected and used material with few market impurities as the raw material for melting, and the adjusted the amount of intra steel impurities.

[0053]

[Table 1]

(7)

Example of this Invention															Comparative Example														
No.	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	Ti	V	W	Al	その他	C+N	δ(C+N)	25(C+N)	Cr											
1	0.0025	0.08	0.08	0.012	0.0008	0.006	0.003	17.6	0.82	0.10	0.007	0.005	0.012		0.0075	0.0450	0.1875	20.06											
2	0.0018	0.12	0.08	0.010	0.0008	0.008	0.004	18.3	0.82	0.10	0.008	0.005	0.010		0.0068	0.0408	0.1700	20.76											
3	0.0018	0.16	0.06	0.011	0.0006	0.006	0.003	19.5	1.20	0.11	0.006	0.005	0.008		0.0069	0.0414	0.1725	23.10											
4	0.0022	0.15	0.09	0.012	0.0009	0.005	0.005	20.5	1.81	0.10	0.008	0.006	0.011		0.0082	0.0492	0.2050	25.93											
5	0.0013	0.10	0.10	0.011	0.0008	0.008	0.004	25.8	0.98	0.12	0.007	0.005	0.010		0.0083	0.0378	0.1575	26.54											
6	0.0020	0.08	0.08	0.012	0.0009	0.005	0.005	18.5	0.52	0.11	0.006	0.004	0.011		0.0050	0.0360	0.1500	20.08											
7	0.0018	0.08	0.08	0.010	0.0006	0.005	0.005	19.2	1.82	0.11	0.008	0.006	0.012		0.0078	0.0468	0.1850	24.60											
8	0.0018	0.10	0.08	0.011	0.0007	0.005	0.004	19.5	2.82	0.11	0.007	0.005	0.010		0.0063	0.0396	0.1650	27.98											
9	0.0012	0.09	0.10	0.012	0.0008	0.006	0.003	29.2	4.02	0.11	0.005	0.005	0.013		0.0062	0.0372	0.1550	41.26											
10	0.0026	0.11	0.09	0.010	0.0008	0.013	0.003	19.2	1.88	0.10	0.008	0.005	0.012	La:0.08	0.0076	0.0456	0.1800	24.84											
11	0.0025	0.10	0.10	0.009	0.0009	0.012	0.012	19.2	2.02	-	0.006	0.006	0.010		0.0085	0.0510	0.2125	25.26											
12	0.0016	0.08	0.06	0.007	0.0007	0.012	0.015	19.0	0.82	-	0.005	0.005	0.011		0.0066	0.0396	0.1650	21.46											
13	0.0015	0.08	0.08	0.006	0.0003	0.011	0.015	19.3	1.95	-	0.008	0.005	0.012		0.0065	0.0390	0.1625	25.15											
14	0.0015	0.06	0.08	0.006	0.0006	0.013	0.016	18.9	2.86	-	0.008	0.005	0.013		0.0065	0.0390	0.1625	27.48											
15	0.0022	0.32	0.43	0.008	0.0005	0.010	0.012	22.3	1.82	-	0.003	0.003	0.010		0.0052	0.0312	0.1300	27.76											
16	0.0025	0.08	0.08	0.007	0.0070*	0.010	0.011	16.8	-	-	0.120	0.008	0.010		0.0105	0.0630	0.2625	16.80											
17	0.0022	0.20	0.09	0.007	0.0075*	0.010	0.008	16.5	-	-	0.120	0.007	0.008		0.0082	0.0552	0.2300	16.50											
18	0.0020	0.06	0.45	0.008	0.0065*	0.009	0.009	18.8	-	-	0.110	0.007	0.010		0.0090	0.0540	0.2350	16.80											
19	0.0016	0.06	0.07	0.020*	0.0068*	0.010	0.010	16.8	-	-	0.130	0.008	0.008		0.0076	0.0456	0.1900	16.80											
20	0.0015	0.30	0.45	0.028*	0.0065*	0.150	0.120	16.7	-	-	0.110	0.007	0.010		0.0085	0.0510	0.2125	18.70											
21	0.0038	0.05	0.08	0.007	0.0058*	0.750*	-	18.2	-	-	0.110	0.005	0.008		0.0088	0.0528	0.2200	19.20											
22	0.0041	0.08	0.08	0.008	0.0065*	-	0.152	19.0	-	-	0.110	0.000	0.009		0.0101	0.0608	0.3525	19.0											
23	0.0200	0.05	0.06	0.006	0.0072*	-	-	19.2	1.80	-	0.120	0.009	0.018		0.0290	0.1740	0.7250	24.6											
24	0.0250	0.45	0.50	0.028*	0.0085*	0.120	0.080	11.5	0.02	-	0.110	0.020	0.002	Nb:0.48	0.0450	0.2700	1.1250	11.56											
25	0.0280	0.25	0.45	0.030*	0.0075*	0.120	0.100	16.8	0.02	-	0.250*	0.023	0.003		0.0510	0.3080	1.2750	10.96											
26	0.0580	0.34	1.20	0.030*	0.0061*	0.230*	8.210*	18.5	0.08	-	0.080	0.031	0.002		0.0590	0.5340	2.2250	18.74											
27	0.0130	0.62	1.00	0.028*	0.0062*	0.240*	12.30*	17.5	2.24	-	0.070	0.023	0.003		0.036	0.2160	0.9000	24.22											
28	"	"	"	"	"	"	"	"	"	-	"	"	"		"	"	"	"											
29	0.0230	0.24	0.53	0.025	0.0230	0.080	0.020	10.1	-	-	0.030	0.005	0.002		0.028	0.168	0.7000	10.10											

*Indicates outside of the range established by this invention.

[0054] Ingots in which the ingot-casted cross-section is round, after being heated for three hours at 1230°C in air, are hot forged by a press method forging machine, and then each ingot is finished on a test slab of the following two types.

[0055] ① Thickness 30 mm, Width 100 mm, Length 120 mm

② Thickness 70 mm, Width 380 mm, Length 550 mm

The slab in ① is assumed to be hot-rolled into a steel sheet of 6 mm thickness, and then cooled while wrapped in heat insulating material that simulates temperature history immediately after the completion of hot-rolling in mass production. Then it is supplied to a Charpy impact test in order to find the tenacity of the hot-rolled coil at room temperature. The specimen is assumed to be a JIS Z2202 No. 4 half size.

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With the slab in ②, the slab surface is cut by machine, the oxidized scale of the surface is removed, and then finished on a slab of 62 mm thickness. This slab is heated to 1200°C in air, and after being finished to a 4 mm thickness by hot rolling, it is annealed while wrapped in heat insulating material that simulates temperature history

- ① No. 25 hot-rolled steel sheet in Table 1 830°C
- ② No. 11~15, 16~23 hot-rolled steel sheet in Table 1 900°C
- ③ No. 1~10 hot-rolled steel sheet in Table 1 930°C
- ④ No. 24 and 29 hot-rolled steel sheets in Table 1 980°C
- ⑤ No. 26 and 28 hot-rolled steel sheets in Table 1 1080°C

At each temperature, re-crystallization progressed, and the temperature at which intermetallic compounds dissolve was assumed. In-furnace time was about 20 minutes.

[0057] Next, the solution annealed hot-rolled steel sheet was finished at a 0.3 mm thickness without cold rolling, while using a multistage Sendzimir-type rolling mill and holding the intermediate annealing midway. The final finishing annealing is performed in a bright annealing furnace for a hydrogen atmosphere in which the dew point is less than -50°C, with the temperature assumed to be the same as the annealing temperature for hot-rolled material. The holding time was one minute, while the in-furnace time was approximately three minutes.

[0058] From this cold-rolled annealing material, the loading separator for specimens meant for performing evaluations of the passivated film in a simulated ambient separator conditions of the following dimensions, and actual solid polymer-type fuel cells, is manufactured from a press form.

[0059] Furthermore, with comparative example No. 27, after manufacturing samples for simulated ambient conditions and separators, we performed one-sided gold plating of thickness 5 µm.

[0060] Specimens for Simulated Ambient Conditions:

Thickness 0.3 mm, Width 10 mm, Length 10 mm

Separator:

Thickness 0.3 mm, Vertical 80 mm, Horizontal 80 mm

Gas Channel: Height 0.8 mm, Peak-to-Peak Gap 1.2 mm

immediately after the completion of hot-rolling in mass production, the same as in ①.

[0056] We performed solution annealing maintained for four minutes at the following temperatures corresponding to the chemical composition, and then forced air cooled.

(Corrugated Processing) These surfaces were mechanically shot ground finished by using SiC abrasive for shot working, ultrasonically cleaned for 15 minutes at 5% HNO₃ + 3% HG and 40°C, alkali spray degreased immediately before testing by using 6% aqueous sodium hydroxide solution, and then, after a simple cleaning with water, soak washed three times with distilled water in a batch-type water tank, and then after further cleaning with distilled water spray for four minutes and drying with a cold blast dryer, were supplied to each test.

[0061] In the simulated ambient conditions tests, the temperature of 2.6 pH aqueous sulfuric acid solution was

raised to 80°C when at a temperature of 25°C reached by using guaranteed reagent sulfuric acid, samples were soaked for six hours in this solution, and then, in addition to evaluating the presence of passivation from the mass loss, the hydrogen bubble generation from the material surface and discoloration of the test solution, in order to more carefully examine the level of metal elution, we measured the passivation maintenance current density at 0.2V vs. SCE.

[0062] Charpy impact test results and the results of tests in simulated ambient separator conditions were as shown in Table 2.

[0063]

[Table 2]

No.	Evaluation in 2.6 pH Aqueous Sulfuric Acid Solution at 25°C		Evaluation according to Loading of a Single Solid Electrolyte-Type Fuel Cell	Impact Value at the Normal Temperature (25°C) for a Hot-Rolled Steel Sheet Air-Cooled after Pressing (Specimen: JIS-4 Half Size (kgf-m/cm ²))	Notes
	Presence of Passivation	Passivation Maintenance Current Density at 0.2V vs. SCE ($\mu\text{A}/\text{cm}^2$)	Rate of Voltage Decrease for a Single Cell after One Hour Elapse 1-(Voltage V after Elapse/Initial Voltage v)		
1	Yes	10~20	<0.05	>15	Example of this Invention
2	"	20~30	<0.05	>15	
3	"	10~20	<0.05	>15	
4	"	10~20	<0.05	>15	
5	"	10~20	<0.05	>15	
6	"	10~20	<0.05	>15	
7	"	<10	<0.05	>15	
8	"	<10	<0.05	>15	
9	"	<10	<0.05	>15	
10	"	10~20	<0.05	>15	
11	"	10~20	<0.05	>15	
12	"	<10	<0.05	>15	
13	"	<10	<0.05	>15	
14	"	<10	<0.05	>15	
15	"	<10	<0.05	>15	
16	No	50~70	0.3~50	2	Comparative Example
17	"	50~70	0.3~0.5	2	
18	"	50~70	0.3~0.5	3	
19	"	50~70	0.3~0.5	2	
20	"	50~70	0.3~0.8	2	
21	Yes	50~70	0.3~0.5	3	
22	"	50~70	0.3~0.5	3	
23	"	50~70	0.5~0.7	1	
24	"	>1mA/cm ²	>0.8	12	
25	"	50~70	>0.8	12	
26	"	10~20	>0.8	25	
27	"	10~20	>0.8	28	
28	"	<10	<0.05	28	
29	"	>1mA/cm ² 50~70	>0.8	18	

[0064] As clearly shown in Table 2, this invention is in a state of passivation within a 2.6 sulfuric acid solution at a temperature of 80°C up from 25°C, with the "passivation maintenance current density" that indicates the level of elution also less than 20 $\mu\text{A}/\text{cm}^2$.

[0065] It has not been said that it is desirable to assume a passivation maintenance current density at as low a level as possible, when stainless steel is used as a separator for a solid polymer-type fuel cell. It is important to be stable and achieve a low level, and although less than 10 $\mu\text{A}/\text{cm}^2$ is the most desirable, the next desirable is 10~20 $\mu\text{A}/\text{cm}^2$.

[0066] The passivation current density in the comparative example is 30~80 $\mu\text{A}/\text{cm}^2$, and although it can be said to be controlled, it indicates that it is in a state in which comparatively large elution from the separator is taking place.

[0067] The inventors determined that performance is insufficient at passivation maintenance current density > 50 $\mu\text{A}/\text{cm}^2$ from the relationship with the performance characteristics of an actual single cell battery as the standard for determining the applicability of a fuel cell separator material. With materials for which passivation maintenance current density is less than 50 $\mu\text{A}/\text{cm}^2$, even evaluation tests for a single actual cell have not been confirming successive performance deterioration at a level that is a problem, and have been determining the high appropriateness of this as an evaluation condition for rapid

simulated ambient conditions. Even in these results, the case of the invention example is at the same level as gold plated materials that are one of the most desirable metal materials in solid polymer-type fuel cell environments (sample steel 27), and it has been determined that their relatively good performance can be insured.

[0068] Next, we evaluated the characteristics while in a loaded state as a separator inside of an actual solid polymer-type single cell battery, measured the voltage of the single cell battery after one hour elapsed from injection of fuel gas into the battery interior, and then examined the rate of voltage decrease according to comparison with the initial voltage. Furthermore, the rate of decrease was obtained according to 1 - (Voltage V after One Hour Elapse/Initial Voltage v).

[0069] The solid polymer-type single fuel cell battery used for evaluation was a reworked commercial battery cell FC50 made by ElectroChem, Inc. of the U.S.A.

[0070] We used 99.9999% hydrogen gas as the anode electrode side fuel gas, and air as the cathode electrode side gas. In addition to the battery body heating the entire body to $78 \pm 2^\circ\text{C}$, it adjusted the humidity control of the battery interior on the side that the exhaust gas water concentration measurement for the cell output side was originally placed. The pressure of the battery interior is 1 atmosphere. The pressure of gas introduced into the battery such as hydrogen gas and air was adjusted at

0.4~0.20 bar. For cell performance evaluation, successive measurements were performed according to conditions in which it was possible to confirm 500 mA/cm²-0.62V at single cell voltage.

[0071] As for the single cell performance measurement system, we reworked and used a fuel cell measurement system that assumed the 890 Series made by Scribner of the U.S.A. as the base. It is possible to predict changes in characteristics according to battery operating conditions, but this is a comparative evaluation under the same conditions.

[0072] Results are shown in Table 2.

[0073] As clearly shown in Table 2, in this invention, the rate of voltage decrease is overall less than 0.05, and becomes the same as with a separator with high corrosion-resistant gold plating at a No. 27 high cost. In addition, in the comparative example that deviates from the chemical composition established in this invention, the rate of voltage drop was quite large at 0.3~0.8.

[0074] In one part of the specimens, an evaluation using long-term tests was implemented, but results that approximately correlated with the short-term test results shown in Table 2 were achieved.

[0075] With regards to the tenacity of stainless steel at normal temperature, ferrite stainless steel is generally inferior in comparison to austenitic stainless steel. However, as clearly shown by the Table 2 Charpy test results, the intra steel C and N content is high, and, in

comparison to the case of the comparative example, the case of the series of invention examples with low C and N content has considerably excellent tenacity. In general, normal temperature tenacity will apparently improve when the sheet thickness thins, so if it is at the level in the case of this invention, there will be absolutely no problems in practical use. Specifically, it can be said that the normal temperature tenacity of a hot-rolled coil that becomes a problem when manufacturing high purity ferrite stainless steel is good. In general, normal temperature tenacity will apparently improve when the sheet thickness thins, so if it is at the level of the steel in this invention, there will be absolutely no problems in practical use.

[0076]

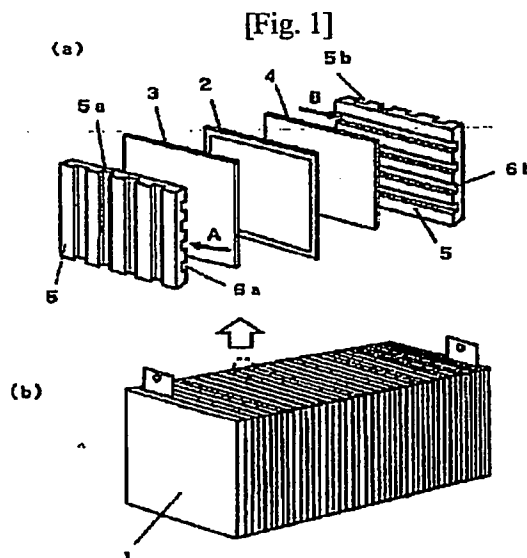
[Effects of Invention] A solid polymer-type fuel cell of this invention has a separator that is composed of ferrite stainless steel, and does not require high-cost gold plating, and so it can be manufactured at low cost.

[Brief Description of Drawings]

[Fig. 1] Drawing showing the construction of a solid polymer-type fuel cell.

[Explanation of Numbers in Drawing]

1	Fuel-Cell
2	Solid Polymer Electrolyte Membrane
3	Fuel Electrode Membrane
4	Oxide Electrode Membrane
5a, 5b	Separator



Continued from the Front

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F Term (Reference)

SH026 AA06 CC05 CX04 EE08 HH05

Review/Edit of Laid-Open Japanese Patent Publication 2000-294256 (特開 2000-293256)

Translator's Forward: In the interests of clarity and my own quality control, I have included the original Japanese text (per the Patent & Utility Model Gazette Database of the Japan Patent Office Website*, which has been verified as matching pdf copies of document provided), the old translation previous provided by myself, and a new translation (which I initially completed without referencing the old translation.

Original Japanese:

【0005】上記の各種の燃料電池は、『燃料電池』と言う共通の呼称で呼ばれているものの、それぞれの電池構成材料を考える場合には、全く別物として捉えることが必要である。使用される電解質による構成材料の腐食の有無、380℃付近から顕在化し始める高温酸化の有無、電解質の昇華と再析出、凝結の有無等により求められる性能、特に耐食性能が、それぞれの燃料電池で全く異なるためである。実際、使用されている材料も様々であり、黒鉛系素材から、Niクラッド材、高合金、ステンレス鋼と多様である。

Old Translation:

[0005] With each of the aforementioned types of fuel cells, in cases when we think about the individual constituent materials of items that are referred to by the common name of "fuel cell," it is necessary for them to be batched as completely different things. This is because the performance obtained by the absence of constituent materials corroding due to the electrolytes used, the absence of high-temperature oxidation that starts to actualize from around 380°C, and the absence of sublimation, reprecipitation, and coagulation of electrolytes, especially corrosion-resistance performance, is completely different for each fuel cell. Actually, the materials used are many, varying from graphite material to Ni clad material, high alloy and stainless steel.

New Translation:

[0005]: Although the aforementioned various types of fuel cells are referred to by the common name of "fuel cell", when taking into account the constituent materials of each cell, they need to be regarded as completely different things. This is because the presence of corrosion of constituent materials due to the electrolyte used, the presence of high temperature oxidation which begins to actualize from around 380°C, the sublimation and redeposition of electrolyte, and the performance demanded by the presence of coagulation, etc., particularly anti-corrosion resistance, is completely different for each fuel cell. In actuality, the materials used are various, ranging from graphite materials, to Ni-clad material, high alloys, and stainless steel.

Notes:

1. First line of clause includes the idiomatic usage of a word that literally means "to catch". Originally translated as "batched"; I have now chosen "regarded".
2. Replaced use of the word "absence" with the more accurate "presence". Original text uses a word that literally means "absence and/or presence", however, "presence" or "existence" is the better term. Original translation due to my level of experience at time of project.
3. More accurate translation of the latter half of the second sentence due to current better understanding of and experience with the terminology involved ("sublimation", "redeposition", "performance", etc.).

※ <http://www.ipdl.inpit.go.jp/Tokujiu/tjsogodb.ipdl?N0000=101>

Original Japanese:

【 0 0 0 6 】 商用化されているリン酸型燃料電池、溶融炭酸塩型燃料電池に使用されている材料を、固体高分子質型燃料電池の構成材料に適用することは全く考えることができない。

Old Translation:

[0006] It is not at all possible to consider the application of material used in commercialized phosphoric acid-type fuel cells and fused carbonate-type fuel cells in the constituent material of solid polymer-type fuel cells.

New Translation:

[0006]: It is completely unthinkable to utilize the materials used in commercialized phosphoric acid fuel cells and molten carbonate fuel cells as the constituent material for polymer electrolyte fuel cells.

Notes:

1. I have chosen a smoother phrasing of the clause; a result of current level of translation experience.
2. The inclusion of "-type" in the names of fuel cells appears in some references but not all. I have not used it in the new translation.
3. Two of the types of fuel cells referenced - "molten carbonate fuel cells" and "polymer electrolyte fuel cells" - were originally translated as "fused carbonate-type fuel cells" and "solid polymer-type fuel cells". ~~The terms used in the old translation were literal translations of the Japanese terminology.~~
The difference in translation between old and new is due to my current level of translation experience, as well as current knowledge of and access to information resources on the technology in question.



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CERTIFICATE OF TRANSLATION

May 25, 2007

I, Dwight Sora, hereby certify that I am competent in both English and Japanese languages.

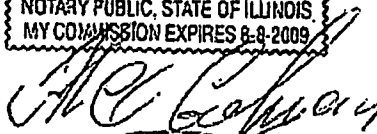
I further certify that under penalty of perjury revised translation of articles [0005] and [0006] of the aforementioned patent document:

[Patent 2000-294256]

from the Japanese language into the English language is accurate and correct to the best of my knowledge and proficiency.


Professional Translator




05.25.2007

X. RELATED PROCEEDINGS APPENDIX

The content of this Related Proceedings Appendix includes the following:

- a copy of the Remarks for Pre-Appeal Conference filed on December 5, 2008; and
- the Notice of Panel Decision from Pre-Appeal Brief Review mailed December 23, 2008.




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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/602,945	06/24/2003	John F. Grubb	RL-1627DIV	1816
<div>7590 12/23/2008 ALLEGHENY TECHNOLOGIES INCORPORATED 1000 Six PPG Place Pittsburgh, PA 15222-5479</div>			<div>EXAMINER DOVE, TRACY MAE</div>	
			<div>ART UNIT 1795</div>	<div>PAPER NUMBER</div>
			<div>MAIL DATE 12/23/2008</div>	<div>DELIVERY MODE PAPER</div>

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Application Number 	Application/Control No. 10/602,945 Tracy Dove	Applicant(s)/Patent under Reexamination GRUBB, JOHN F. Art Unit 1795
Document Code - AP.PRE.DEC		

Notice of Panel Decision from Pre-Appeal Brief Review



This is in response to the Pre-Appeal Brief Request for Review filed 12/5/2008.

1. ☐ **Improper Request** – The Request is improper and a conference will not be held for the following reason(s):

- ☐ The Notice of Appeal has not been filed concurrent with the Pre-Appeal Brief Request.
- ☐ The request does not include reasons why a review is appropriate.
- ☐ A proposed amendment is included with the Pre-Appeal Brief request.
- ☐ Other:

The time period for filing a response continues to run from the receipt date of the Notice of Appeal or from the mail date of the last Office communication, if no Notice of Appeal has been received.

2. ☒ **Proceed to Board of Patent Appeals and Interferences** – A Pre-Appeal Brief conference has been held. The application remains under appeal because there is at least one actual issue for appeal. Applicant is required to submit an appeal brief in accordance with 37 CFR 41.37. The time period for filing an appeal brief will be reset to be one month from mailing this decision, or the balance of the two-month time period running from the receipt of the notice of appeal, whichever is greater. Further, the time period for filing of the appeal brief is extendible under 37 CFR 1.136 based upon the mail date of this decision or the receipt date of the notice of appeal, as applicable.

☒ The panel has determined the status of the claim(s) is as follows:

Claim(s) allowed: _____.

Claim(s) objected to: _____.

Claim(s) rejected: 38-40, 42-43.

Claim(s) withdrawn from consideration: _____.

3. ☐ **Allowable application** – A conference has been held. The rejection is withdrawn and a Notice of Allowance will be mailed. Prosecution on the merits remains closed. No further action is required by applicant at this time.

4. ☐ **Reopen Prosecution** – A conference has been held. The rejection is withdrawn and a new Office action will be mailed. No further action is required by applicant at this time.

All participants:

(1) JENNIFER K. MICHENER

(2) Tracy Dove

(3) Dah-Wei Yuan

(4) _____



Serial No. 10/602,945
Attorney Docket No. RL-1627DIV

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of John F. Grubb	:	
	:	
Group Art Unit 1795	:	FERRITIC STAINLESS STEEL
	:	HAVING HIGH TEMPERATURE
Serial No. 10/602,945	:	CREEP RESISTANCE
	:	
Filed June 24, 2003	:	
	:	Confirmation No. 1816
Examiner Tracy Mae Dove	:	

REMARKS FOR PRE-APPEAL BRIEF CONFERENCE

Sir:

In response to the June 5, 2008 final Office Action ("the Final Office Action") issued in the above application, Applicant requests a Pre-Appeal Brief Conference in accordance with guidelines in the "New Pre-Appeal Brief Conference Pilot Program" announcement appearing in the 12 July 2005 *Official Gazette*. In accordance with those guidelines, arguments for the conference are presented herein. Submitted concurrently herewith are: a Pre-Appeal Brief Request for Review (form PTO/SB/33); a Notice of Appeal; the fee under 37 CFR § 41.20(b)(1); and a Petition for Extension of Time and associated fee. Any deficiency or overpayment may be charged or credited, respectively, to Deposit Account No. 11-1110. The issues addressed herein are ripe for appeal in accordance with 37 CFR § 41.31(a)(1), and the claims have been subject to at least two rejections.

Arguments

In the Final Office Action, the Examiner rejects each of pending claims 36-40, 42, and 43 as being unpatentable under 35 USC § 103(a) over U.S. Patent No. 6,613,468 to Simpkins et al. ("Simpkins") in view of JP 2000-294256 to Taruya et al. ("Taruya"), "as evidenced by" U.S. Patent No. 5,424,144 to Woods ("Woods"). Applicant submits that the invention recited in the claims under examination does not result and would not have been suggested by the asserted combination of Simpkins, Taruya, and Woods. Applicant also submits that the record evidence of unexpected results confirms that the

claimed invention would not have been obvious. For at least those reasons, the rejections should be withdrawn and the claims allowed.

As discussed in Applicant's August 9, 2007 Response to Office Action, Taruya explains that the components of SOFCs (including interconnects) are not interchangeable with the same components from other types of fuel cells because of wide differences in fuel cell operating conditions. For example, paragraphs 0005 and 0006 of Taruya (emphases added) read as follows:

[0005]: Although the aforementioned various types of fuel cells are referred to by the common name of "fuel cell", when taking into account the constituent materials of each cell, they need to be regarded as completely different things. This is because the presence of corrosion of constituent materials due to the electrolyte used, the presence of high temperature oxidation which begins to actualize from around 380°C, the sublimation and redeposition of electrolyte, and the performance demanded by the presence of coagulation, etc., particularly anti-corrosion resistance, is completely different for each fuel cell. In actuality, the materials used are various, ranging from graphite materials, to Ni-clad material, high alloys, and stainless steel.

[0006]: It is completely unthinkable to utilize the materials used in commercialized phosphoric acid fuel cells and molten carbonate fuel cells as the constituent material for polymer electrolyte fuel cells.

The above passage pertains to all other constituent materials of a fuel cell (including the interconnect) because of (due to) the electrolyte. The choice of electrolyte drives the conditions, temperature and corrosion especially, in the fuel cell, which affects these other constituents. Taruya explains that it is "completely unthinkable" to use materials (including interconnects) from other kinds of fuel cells in polymer electrolyte fuel cells. One skilled in the art, after considering Taruya, would not have been motivated to use, and indeed would have been taught not to use, any of the constituent materials of the fuel cell described in Taruya in another type of fuel cell, especially one operating at a much higher temperature.

The Examiner cites Woods as showing "a separator suitable for use in various known types of fuel cells, such as solid oxide fuel cells and polymer electrolyte fuel cells." In essence, the Examiner attempts to disprove what is taught by Taruya regarding interchangeability of fuel cell components by citing to Woods. Any conflict between Taruya and Woods on this issue, at best, is evidence of the unsettled nature of the issue. The existence of any such conflict between the cited references is not sufficiently definitive to constitute a teaching, contradictory to Taruya, that "...the

interconnect (for a solid polymer fuel cell) of Taruya could have been used the interconnect (solid oxide fuel cell) of Simpkins", as the Examiner contends. Although Woods' column 1 (to which the Examiner cites) does refer to some use of ferrous metal separator plates, it also refers to disadvantages of using ferrous metals in molten carbonate fuel cells:

In molten carbonate fuel cells, in particular, under fuel cell operating conditions, in the range of about 500°C to about 700°C, molten carbonate electrolytes are very corrosive to ferrous metals which, due to their strength, are required for fuel cell housings and separator plates. The high temperature operation of stacks of molten carbonate fuel cells increases both the corrosion and thermal stress problems in the wet seal area, especially when the thermal coefficients of expansion of adjacent materials are different.

One having ordinary skill in the art faced with the teachings of Taruya and contradictory teachings derived from Woods would not have been motivated to utilize the material of Taruya in the solid oxide fuel cell of Simpkins with any expectation that the interchange of materials would be successful. For at least this reason, the asserted combination of references fails to teach or suggest the invention recited in claim 36 of the Subject Application.

Applicant discussed record evidence of unexpected results in, for example, Applicant's February 29, 2008 Response to Office Action. Pages 5-8 of that response discuss record evidence clearly showing that the range of Nb, Ti, and Ta in the alloy composition recited in independent claim 36, which is $0.5 \leq (\%Nb + \%Ti + \frac{1}{2}(\%Ta)) \leq 1$ (the "Recited Range"), provides properties that are unexpected and surprising relative to compositions including a combined concentration of the three elements that is outside the recited range. As discussed in the Subject Application, six Nb-containing heats of ferritic stainless steel with the compositions set out in the table on page 21 of the specification ("the Table") were prepared. The heats' Nb concentrations were varied to provide heats falling within (heats WC71, 73, 74, and 75) and outside (heats WC70 and 72) the Recited Range. Relatively small increases in Nb concentration provided significantly and surprisingly improved creep resistance at SOFC operating temperatures, as well as significant improvements in microstructural stability and certain other mechanical properties.

As discussed on pages 6-8 of the February 29 response and on pages 28-39 of the specification, tested alloy compositions in the Table satisfying the Recited Range exhibited significantly improved creep resistance, which was especially significant at higher test temperatures. For example, alloys satisfying the Recited Range exhibited creep rupture strength of 400 hours at 1000 psi and 900°C, while the conventional alloys tested exhibited creep rupture strength of only 156 hours at lower pressure (900 psi). Alloys listed in the above table having compositions satisfying the Recited Range exhibited a time to 1% creep strain of at least 100 hours at 900°C under a load of 1000 psi. In contrast, the conventional alloys evaluated exhibited a time to 1% creep strain of only 2.5 hours at 900°C under a load of 900 psi. Also, the tested alloys satisfying the Recited Range exhibited a time to 2% creep strain of at least 200 hours at 900°C under load of 1000 psi. In contrast, the conventional alloys evaluated exhibited a time to 2% creep strain of only 5 hours at 900°C under load of 900 psi.

Pages 24-26 of the Subject Application's specification discuss the improvements in microstructural stability of the heats in the Table including at least 0.5% Nb. The alloy compositions in the Table that do not satisfy the Recited Range (WC70 and 72) readily recrystallized at 1750°F and also experienced significant grain growth at temperatures of about 1950°F and above. In contrast, the alloys in the Table satisfying the Recited Range did not show any evidence of recrystallization until heated to the substantially higher temperature of about 2000°F. Pages 27-28 of the specification as originally filed describe the unexpected and significant improvements in yield strength, tensile strength and hardness exhibited by the alloys in the Table that satisfy the Recited Range.

Given the inventor's understanding of the mechanism that may be responsible for the unexpected and substantial improvement in alloy properties resulting from increases in Nb concentration, the inventor further concluded that Ti and/or Ta may be included in the alloy, either along with or in place of Nb, in a combined concentration that falls within the Recited Range to achieve similarly significant improvements in the indicated alloy properties.

Therefore, adjusting the composition of a ferritic stainless steel alloy to satisfy the Recited Range provided a significant and unexpected improvement in several properties

Therefore, adjusting the composition of a ferritic stainless steel alloy to satisfy the Recited Range provided a significant and unexpected improvement in several properties including creep properties, microstructural stability, yield strength, tensile strength, and hardness. These significant improvements yield a ferritic stainless steel suitable for use as interconnect materials in SOFCs, which provides many advantages over conventional ceramic interconnect materials.

Thus, the asserted combination of references would not have disclosed or suggested the claimed invention. The Subject Application also clearly shows that alloy compositions satisfying the Recited Range exhibit unexpected, surprising, and significant improvement in several properties, including properties critical to materials used in SOFC interconnect applications. That the Recited Range results in these unexpected, surprising, and significant improvements clearly rebuts any *prima facie* case that may have been established by the cited combination of references.

Conclusion

Applicants respectfully requests that the claim rejections in the Final Office Action be withdrawn and that all claims under examination in the Subject Application be allowed.

Respectfully submitted,



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Date: December 5, 2008

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XI. CONCLUSION

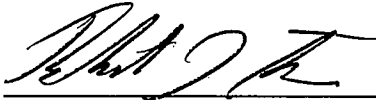
For the reasons discussed above, Appellant respectfully asks the Board to direct the Examiner to: (1) withdraw the obviousness rejections under 35 U.S.C. § 103(a) in the Final Office Action and (2) issue a Notice of Allowance for the claims pending in the Subject Application.

July 6, 2009
Date

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